ASSESSMENT OF SPONTANEOUS HEATING SUSCEPTIBILITY OF INDIAN COALS BY EXPERIMENTAL TECHNIQUES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

B.TECH AND M.TECH DUAL DEGREE

IN

MINING ENGINEERING

BY

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DEPARTMENT OF MINING ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
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CERTIFICATE

This is to certify that the thesis entitled "**Assessment of Spontaneous Heating Susceptibility of Indian Coals by Experimental Techniques**" submitted by **Sri Bhaskara Behera** in partial fulfilment of the requirements for the award of B.Tech And M.Tech Dual Degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my guidance and supervision.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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Date:

ABSTRACT

Introduction

Coal mine fires due to spontaneous heating are a major problem in coal mining industry worldwide. In this process, coal reacts with oxygen of air at ambient temperature liberating heat, which if not properly dissipated may lead to increase in temperature, which in turn enhances the rate of reaction, ultimately culminating in open fire. Occurrences of spontaneous heating in mines are major cause of accidents, resulting in loss of lives and property. Development of spontaneous heating in any section/part of a mine creates problems for the workings of nearby areas. They also cause loss of coal reserves, sterilisation of resources, reduction of heating values and coking properties, and environmental pollution. Since, all coals do not show the same heating tendency on exposure to air, its assessment is essential to plan the production and storage capabilities in mines. Different experimental techniques are followed in different countries for assessment of the spontaneous heating tendency. However, any particular experiment does not yield a confirmatory result. Therefore, some researchers suggest to conduct multiple number of experiments in order to reach at a definite conclusion (Banerjee, 2000). In this study, an attempt has been made to study the spontaneous heating tendency of Indian coal seams by using different experimental techniques.

Experimental Investigation

In order to assess the spontaneous heating tendency of Indian coals, 48 coal samples were collected from all major coalfields of the country. An additional sample from New South Wales, Australia; has also been used for comparison. The intrinsic properties of these samples were determined using proximate analysis and bomb calorimeter. The mineralogical composition were determined by XRD analysis. The spontaneous combustion susceptibility was determined by using Differential Thermal Analysis (DTA) and Fourier transform infrared spectroscopy (FTIR). The indices that were obtained from DTA analysis are transition Temperature (Tc), Slopes of IIA, IIB and overall of stage II obtained from DTA thermogram. Similarly the aliphaticity of the coal samples were obtained from FTIR study. The correlation study was carried out between intrinsic parameters and susceptibility indices in order to assess their acceptability.

Results and Discussion

The proximate analysis of the coal samples showed that the moisture content of coal samples varied from 0.6% to 37.30 %, volatile matter content from 10.80% to 43.03% and ash content 3.40% to 48.51%. Gross calorific value of the coal samples varied from 2366.66 cal/gm to 8075.08cal/gm. Thus, the coal samples cover a broad spectrum.

The transition temperature (T_c) value of the coal samples varied from 115.39^0C to 238.44^0C . Panigrahi and Sahu (2004) have found that the coal seams having onset temperature in the range of 122^0C to 140^0C are highly susceptible to spontaneous heating. As per their sample SCCL-7 is

the highest susceptible to spontaneous heating. This is followed by SCCL-6 and MCL-4 with a T_c of 151.70^0 C and 152.74^0C respectively. Similarly the samples collected from ECL have lower susceptibility to spontaneous heating with high transition temperature value. Similar results were obtained from IIA and IIB slopes obtained from DTA thermogram.

The aliphaticity obtained from FTIR study varied from 0.73 for ECL-1 to 2.37 for SCCL-7. This indicates that the coal structure of SCCL-7 contains a high number of aliphatic bonds as compared to aromatic bonds and makes it to be highly susceptible to spontaneous heating. From the mineralogical study through XRD analysis it has been seen that most of the mineral that are present in the coal sample are silica based mineral i.e quartz and kaolinite followed by muscovite and orthoclase. Sample from NEC, NLC, NCL and MCL showed the presence of some sulphur associated minerals. The correlation between intrinsic properties and susceptibility indices has been presented below:

Sl. No.	Independent variable	Tc	IIA	IIB	II	Aliphaticity
1.	M	0.62	0.64	0.08	0.46	0.48
2.	VM	0.34	0.58	0.36	0.58	0.08
3.	A	0.19	0.15	0.13	0.23	0.43
4.	FC	0.39	0.61	0.38	0.74	0.70
5.	Calorific value	0.22	0.88	0.24	0.32	

Conclusion

From the experimental investigation it was found that the coal samples from SCCL were very highly susceptible to spontaneous heating, whereas the samples from BCCL and ECL were found to less susceptible to spontaneous heating. The correlation study showed that IIA slope obtained from DTA thermogram could be a better indicator than transition temperature. It is hoped that the outcome of the project work will help the practicing field engineers to plan their workings in advance so that spontaneous heating of coal leading to mine fires can be avoided.

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 General

Mine fires due to spontaneous combustion is a major concern for the coal mining industry across the world. Fires in coal mines could be anthropogenic, induced from nearby fire affected seams or due to spontaneous combustion which is a common phenomenon. All types of coal generally absorb oxygen and oxidise to evolve CO, CO_2, and H_2O along with heat. This heat slowly dissipates into air. If the rate of oxidation is slower and the amount of heat dissipation is higher than the amount of heat accumulation then coal doesn't reach ignition temperature (Critical Temperature for Oxidation) and the process is referred as weathering. But in case the oxidation rate is faster and the amount of heat accumulation is higher than the amount of heat dissipation, it makes the coal reach the critical temperature for oxidation resulting in coal ignition which is referred as spontaneous heating or auto oxidation of coal. Several incidents of mine fires followed by spontaneous combustion of coal can be found since the beginning of coal mining history. It not only causes huge loss of coal resources but also poses a great threat to the environment as well as life of the mine workers.

Coal found in a lot of Indian coalfields is highly susceptible to spontaneous heating. In India, spontaneous combustion is seen in all major coalfields like Raniganj, Jharia, Karanpura, Bokaro, Ib-valley, Talcher etc. (Pattanaik et.al, 2011). An example of most spontaneous combustion susceptible coalfield is Jharia coalfield in BCCL India which has many mine fires burning since 1925 (Ramlu, 2007). Cases of fire have also been reported during transportation of coal. Another problem associated with spontaneous heating is the deterioration of coal quality due to oxidation on coming in contact with air. This reduces the heating value of coal and affects its coking properties. Storage and transport of run-of-mine coal is an integral part of coal mining industry. Coal handling systems are required not only at mines but also at power plants and metallurgical coking plants. Investigators have found that these incidents could have been prevented if proper planning and design for coal production and handling was adopted. This has led to number of researches to investigate and try to understand the process

of spontaneous heating and find methods to prevent it. It is well known that spontaneous heating of coal depends mainly on two types of factors, such as intrinsic and extrinsic. The intrinsic parameters are mainly associated with the nature of the coal, i.e. its physico-chemical characteristics, petrographic distribution and mineral make up. On the other hand, the extrinsic parameters are related to atmospheric, geological and mining conditions prevailing during extraction of coal seams and these are mainly site specific. The intrinsic coal properties that control propensity for self-heating have been the subject of many investigations (Singh and Demirbilek, 1987; Smith and Lazzara, 1987; Beamish, 2008a; Panigrahi and Saxena, 2001. Kucuk et al., 2003).

Scientists have explored the subject from both chemical and physical contemplations and developed many theories trying to explain the process of spontaneous heating. Numerous techniques have been proposed to quantify the weakness of coal to unconstrained heating. Some of these techniques are Crossing Point Temperature in India , Olpinski Index in Poland, Russian U- Index in Russia, Adiabatic calorimetry in U.S.A etc. However, any particular experiment does not yield a confirmatory result. Therefore, some researchers suggest to conduct multiple number of experiments in order to reach at a definite conclusion (Banerjee, 2000). In this study, an attempt has been made to study the spontaneous heating tendency of Indian coal seams by using different experimental techniques and correlate them with thie intrinsic properties.

1.2 Objectives

Keeping the above problem in mind, the work has been planned with the following objectives:

- To understand the mechanism of spontaneous heating of coal.
- Determination of the intrinsic properties of the coal samples by proximate analysis and bomb calorimetry.
- Determination of spontaneous heating susceptibility of coals seams by Differential thermal analysis, Fourier transform infrared spectroscopy and X-Ray diffraction techniques.
- Correlation of spontaneous heating susceptibilities indices with that of the intrinsic properties.

CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

A lot of research has been carried out regarding the spontaneous heating of the coal, but still more research is required to clearly understand the mechanism and correlate it with its intrinsic properties. A summary of some of the relevant literature has been presented below:

Banrjee and Chakravarty (1967) recommended differential temperature Analysis (DTA) for the investigation of spontaneous heating of coal, especially in arranging coals regarding their susceptibility to self-heating. A standard trial system for completing DTA studies had been recommended by them. Calcined alumina is suggested as inactive reference material for DTA tests. A heating rate of 5 ^{0}C/min was suggested for such studies. Common temperatures acquired from different coals are incorporates in this study to account for self-heating phenomenon.

Clemens et al. (1989) dissected the response of low rank coals (from New Zealand) with oxygen at temperatures of 30, 60, 90, 120, 150 and 180^{0}C utilizing differential thermal techniques. They discovered a prompt, sharp exothermic response in the middle of oxygen and coal. It was watched that the exothermicity increases with increasing temperature and is most proclaimed for the coal most inclined to spontaneous combustion. It was inferred that DTA systems may be valuable to acquire data about mechanism involved and susceptibility of coals to unconstrained heating when connected to isothermal low temperature oxidations of coal.

Ndaji (1995) reported FT-IR investigation of oxidation of coal sample. They reported that the proportion of aliphatic to aromatic content of distinctive coals differs from 3.33 to 0.91 and these aliphatic parts diminishes at diverse rates for distinctive segments i.e. the rate of reduction of aliphatic parts with temperature varies from one coal to other, however the oxidation of coal even at low temperatures underneath 2000 C, decreased the aliphatic/aromatic proportion and increase the hydroxyl/aromatic proportion alongside an increase in oxygen containing functional group like C=O, -COOH, and so forth. All these affirm the procedure of oxidation of hydrocarbons in coal with an increment in temperature

leading to loss of aliphatic moiety from the coal structure through thermal and reactant discontinuity leading to increase in the aromatic nature of the coal substance.

Chen et al. (1996) studied the mechanism of ignition of coal of distinctive quality, by thermogravimetry (TG) and differential thermal analysis (DTA). Different tests on coal ignition were completed at a low heating rate (10 K min−1) with molecule sizes extending from 37 to 4000 μm. From the laboratory work they concluded that ignition estimation by TG-DTA is a brilliant strategy for precise determination of the ignition temperature of coal particles.

Cetinkaya (1998) watched the reduction in the peaks of aliphatic C-H because of oxidation prompting increment in C=O peaks in the coal samples. Anyhow, as the temperature stretches around 250^0 C, C=O peaks reflects diminishing pattern with further increment in temperature and as the temperature thus achieves 500-5500 C, peaks practically vanished.

Pis et al. (2003) used differential thermal analysis (DTA) technique to study the self-heating conduct of new and oxidized coals. Oxidation was performed in air at 200°C for times of up to 72 h. Six coals running from a High A Bituminous coal to a semi-anthracite were utilized. As the rank of the coal increases, both the self-heating and the end of combustion temperatures likewise increases. The aggregate heat loss (area under the DTA curve) increased with the rank of the coal. An increment in the self-heating temperature, a decrease in the temperature of the end of combustion and a reduction altogether heating stream were seen as an outcome of coal oxidation. A relationship between the aggregate loss of heat and the calorific esteem was decided utilizing the ASTM standard technique is called pointed out.

Reyes et al. (2003) conducted the characterization and differentiation, utilizing Mössbauer spectroscopy (MS) and X-Ray diffraction (XRD), of coal specimens with distinctive ash remains and sulfur substance acquired in three spots relating at cuts in different coal seams from the Guachinte mine, Valle, Colombia. The mineral stages distinguished by XRD were is normally kaolinite, quartz, pyrite and gypsum, and specifically dolomite and calcite. MS confirms the presence of pyrite and kaolinite, other than confirmations the additional findings of jarosite which was not recognized by the XRD results due their low sums. In the high

mineral matter ash remains test quartz and hematite was distinguished by XRD, the last one affirmed by MS results. A second stage in this example was recognized by Mössbauer spectroscopy, which could be super paramagnetic hematite. Rietveld refinement for XRD design from a specimen is reported.

Saikia et al. (2007) investigated with coal samples gathered from Makum coal field, Assam, India by XRD and FT–IR procedures. The X-Ray diffractogram demonstrated the presence of some crystalline carbons in Assam coals as demonstrated by the presence of peaks. The radial distribution functional (RDF) system was applied for the determination of structural aspects of the coals. The study shows that the coals are lignite in sort and there is no confirmation of graphite-like structures. The greatest in the G(r) plots of function of radial distribution of atoms (FRDA) identifies with distinctive separations between carbon molecules of aliphatic chains. The curve intensity profiles acquired from FRDA show very customary sub-atomic packets for this coal. The coals were discovered to be lignite in nature. FT–IR study demonstrates the vicinity of aliphatic carbon, C=O and C–O extending connected with –OH and –NH extending vibrations. Kaolinite and quartz were likewise discovered to be significant minerals in Assam coals by FTIR spectroscopy. They were of the opinion that the distinction in intensities of carbonyl groups of the coal examples was liable to relate with rank.

Pone et al. (2007) made gas temperature estimations at coal-flame vents range from 34°C to 630°C. The coal-flame gas minerals (CFGM) distinguished included sulfur mixes and salammoniac. X-Ray diffraction (XRD) investigations of CFGM by-items confirmed the presence of mascagnite ((NH4)2SO4), illite ($(Al,Si)_4O_{10}[(OH)_2,H_2O]$), letovicite ($(NH_4)3H(SO_4)_2$), phlogopite ($KMg_3(AlSi_3)O_{10}(F,OH)_2$), titanium dioxide ($TiO_2$), barite ($BaSO_4$), iron sulfate ($FeSO_4$), gypsum ($CaSO_4 \cdot 2H_2O$) and silicate.

Rao et al. (2011) reasoned that with increase in faliphaticity of a sample of coal, the risk towards unconstrained heating likewise increases. For a sample of coal if faliphaticity is high and likewise in the event that it diminishes at a quicker rate with increase in temperature, such a coal is very inclined to spontaneous heating.

Wang et al. (2011) completed an experiment for the time development of the coal structure at low temperature at almost 120^0C for the time of 14 days. The coal characterization was finished with the assistance of FT-IR. It was found that the groups like carboxyl and ether group are primary highlight for expanding the oxidation of the coal. It was discovered the aromatic structures had additionally beneficial outcome on the spontaneous combustion of the coal.

Ejlali et al. (2011) utilized a scientific model to study the self-heating systems of wet permeable media (coal stockpile). Three parameters were utilized to be specific, porosity of the medium, dampness substance and Darcy number. It was watched that the heating happens in three stage. In the first place temperature ascends because of self-heating, then second stage comprises of steady temperature because of fluid to vapour stage change. In third stage temperature again rises and ignition may happen at any moment. It was found that the model can be utilized for estimation of time that a regular stockpile can be kept safe.

Wang et al. (2013) experimented to choose a specific productive inhibitor to retard the sudden ignition of the coal. The examination of the intricate coal structure was finished with the assistance of FT-IR (Fourier Transformation Infrared range). Clarification of the dynamic practical functional groups was embraced to add to a successful system for determination of a proficient chemical inhibitor. The defence of the methodology was checked utilizing the CPT, oxygen utilization, CO emanation rates. The FT-IR investigation was utilized to monitor the dispersion and changing the concentration of dynamic functional groups. The active groups with lower actuation energy were killed by chemical inhibitor to destroy the future oxidation.

Jo et al. (2013) utilized CPT and gas chromatography to relate sudden ignition susceptibility and rank of coal. They utilized three coals – two Indonesian Lignite and one Chinese bituminous coal. FTIR was utilized to break down the hydrocarbon exhibit in coals. It was reasoned that coals of lower rank have more substance that can respond with oxygen at low temperatures making them more vulnerable to sudden ignition than higher positioning coal.

Xia et al. (2014) proposed a spontaneous ignition model of underground mine by setting up an arrangement of halfway differential comparisons, including a coal deformation

mathematical statement, an oxygen stream and transport comparison and a energy conservation mathematical equation. Quantitative assessment of extrinsic and intrinsic elements, for example, coal porousness, difference in pressure, oxygen-utilization rate, and response of heating of coal was done to survey their impact on spontaneous combustion. It was found that at time of coal oxidation, the oxidation fixation has a "S-sort" descending pattern, though the heating temperature of coal and the gas speed show "S-sort" upward patterns. They suggested that the simulated results can give a few recommendations to control of spontaneous heating.

CHAPTER 3

MECHANISM OF SPONTANEOUS HEATING OF COAL

3. MECHANISM OF SPONTANEOUS HEATING OF COAL

3.1 Mechanism of Spontaneous Heating

Coals when exhibited to air absorb oxygen even at temperatures much lower than the average ambient ones and evolve heat. But, all coals do not end-up in a fire due to the fact that they do not reach the temperature at which coal will oxidise in a self-propellant manner, on its way to ignition and fire, which is known as 'Critical Temperature for Oxidation'. It is because of the emission of heat. When this emission of heat is more than accumulation then coal cannot reach to critical temperature, which is known as 'Weathering'. If accumulation is more than emission, the temperature rise of coal reach and crosses the critical temperature with steadily rise in the rate of oxidation in a continuous manner finish up with ignition, termed as Spontaneous Heating of Coal (Fig 3.1). This typical physico-chemical phenomenon includes absorption, adsorption and hence oxidation of the coal, with the oxygen available from the surrounding air.

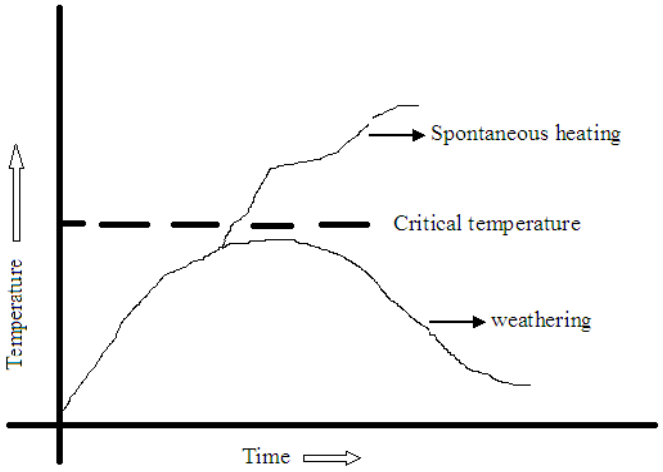


Figure 3.1: Difference between Spontaneous Heating and Weathering

The auto oxidation of coal is a complex physico-chemical process which is accompanied by the absorption of oxygen, formation of coal-oxygen complexes and their decomposition

leading to the liberation of heat. This complexity of the process is enormous because of the great diversity in the coal substance with the associated mineral matter and the conditions of oxidation. During the oxidation of the heterogeneous mass, concurrent and overlapping reactions take place which are very difficult to separate out. The rate of oxidation at ambient temperature gives a measure of the proneness of coal to auto-oxidation. This low temperature aerial oxidation of coal is not a singular chemical process but a complex phenomenon generally comprising of several simultaneous and interesting chemical processes which result both in erosive material removal and structural alteration of the organic matter. A large number of stable chemical chain reaction takes place due to several oxidation states of carbon and a variety of strong carbon oxygen bonds are formed. The observable elemental, compositional and structural changes reveal that the reaction of oxygen with solid coal is a time dependent dynamic process. Observable changes in coal molecular structure and composition arise from prolonged sequence of events whose components exhibits complex inter-relationships. Thus the reaction environment is heterogeneous intrinsically because two bulk phases, solid and gas are present and extrinsically because diverse structural changes induced by the reaction, affect the overall coal reactivity.

Porous solid absorbs the liquids or gases or the solution of gas/liquid, which is known as sorption. When accumulation restricts at the surface liberating heat and rate of penetration is negligible then it is considered as adsorption but if uniform penetration in the bulk of the solid occurs, then it is called absorption. The absorptions is always an endothermic phenomenon and starts from the surface of the solid and consumes heat of the solid for penetration. The energy at the surface always decreases. The process where physical forces like inter-molecular attraction are responsible, are known as physical adsorption or Van der Wall's adsorption, but when operative force needs to break the chemical nature of the compound is called chemisorptions or activated adsorption. This physical adsorption is predominant at very low temperature whereas chemical adsorption is very low at low temp whereas rate of reaction increases with increase in temperature.

Coal gets heated up on absorbing oxygen, whose decomposition phenomenon can be expressed/explained in the following manner. Oxidation is very slow below 50^oC and accelerates above 50^oC, but above 80^oC, a period of steady state is maintained, probably due to the removal of moisture of coal. The removal of oxides of carbon beings from 120^oC. The interaction of oxygen with coal accelerates rapidly upto 180^oC and thermal decomposition starts between 180^oC to 220^oC. Self sustained process of combustion begins in between 220^oC to 275^oC with very rapid rise of temperature until the ignition point is attained.

3.2 IMPORTANT FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

There are mainly three factors which affect the spontaneous heating of coal, viz. geological, seam and mining factors. Role of some of the dominant factors in each category with respect to spontaneous heating has been has been discussed below:

Geological Factors

Caving Characteristics: In mine where partial extraction is practiced, adequate pillars are left to support the superincumbent strata, and the caving characteristics are generally of little significance. In order to reduce the amount of leakage airflow within the extracted area, it is desirable for the waste to be filled with as fine a material as possible. Thus this material occupies the greatest volume and fills the void. The type of friable waste, and sufficient emphasis should be laid on the non-combustible or non-carbonaceous material. In the South African coalfields the inter burden tends to be carbonaceous and, on breaking up to fill up voids, spontaneous heating tends to develop. In a totally caved longwall or in a secondary-extracted bord and pillar section, the caving characteristics of the roof assume great importance, and it is imperative to reduce the amount of leakage overflow within the extracted area. In some coalfields the fallen shale or 'slate' roof fires spontaneously. In that case it is necessary to seal off rooms as soon as they have finished or have been stopped by great falls; otherwise, the material heats and fires quickly. It would be better if the pillars in such mines were extracted, so that there would be a complete closing down of the roof.

Coal Friability: The more friable the coal is, the larger the surface area exposed to oxidation, thus tending to yield more heat per unit volume of coal.

Seam Factors

Rank: The rank of a coal depends on the character of the original plant debris from which it was formed and the amount of change that its organic matter has undergone during the period of formation. And increasing carbon content and with it a decreasing oxygen content are the most commonly accepted criteria of increasing rank. The higher the rank, e.g. anthracite, the slower the oxidation process, whilst lignite of low rank, oxidizes so rapidly that is often stated that is cannot be stored after mining without ignition. There are numerous anomalies to a straight rank order. One part of a seam may be particularly liable to spontaneous combustion, a seam of higher rank may prove more troublesome than one of a lower rank or even the same seams in different mines may react differently.

Petrographic composition: The National Coal Board carried out a series of oxidation tests on hand picked petrographic constituents from five coals ranging from high ranking coking coal to low rank bituminous coal. The results of these tests showed that in all cases fusain was the least reactive, and in general durain was more reactive than vitrain. These results enable calculations to be made of the reaction velocities of vitrinite, exinite and intertinite and showed that above 165^{o}F exinite has a much greater oxidation rate than the other two constituents. Thus it appears that a count of these macerals may be useful, together with rank in determining the susceptibility of coals to spontaneous combustion.

Particle Size: A solid coal face generally presents very little danger of spontaneous combustion, partly due to the small surface area and partly due to the very low permeability of solid coal to gases. It is, however generally when coal is shattered in mining, or broken by roof pressure, or when falls and faulting occur that spontaneous combustion is likely to take place.

Moisture: The effect of moisture on spontaneous heating is uncertain. A small quantity seems to assist rather than retard the heating whilst large quantities of moisture retard the heating. However as in a surface stockpile alternative drying and wetting of the coal accelerates the heating process.

Bacteria: It is observed that thiobaccillus ferro-oxidants and ferro-baccillus thio-oxidants are the bacteria which play the role for auto-oxidation of coal. These bacteria are inactive at -193-^{o}C temp. and +560^{o}C temp.

Pyrite contents: The most commonly occurring sulphides in coal are reported to be pyrite, marcasite and melnikovite-pyrite. In addition to these, most coal seams contain small quantities of sparelite, galena and chalcopyrite. Many factors like temperature, relative humidity, presence of microbial agents and overall mineralogy of coal are important while dealing with the pyrite effect in the low temperature oxidation of coal. Pyrites are having catalytic effect and the mechanism is that the oxidation product accelerates the rate of oxidation of organic compounds in the coal. This pyrite oxidation leads to the formation of Fe^{+3} compounds, which can participate in various redox processes. In the presence of oxygen in air, the reduced Fe^{+2} compounds can easily be reoxidized to Fe^{+3}. This pyrite oxidation leads to swelling, which in turn, can cause breakage of the coal particles leading to an enlarged surface area. Heat caused by this oxidation is the same for coal and pyrite in dry state and that the reactivity of wet coal is only doubled whereas the reactivity of finely dispersed pyrite is raised to tenfold.

Mining Factors

Mining Methods: An advancing method of mining on the longwall system leaves extracted areas lying between the entries serving the working places. The ventilating pressure differences will encourage air to flow across these areas, with the accompanying risk of combustion. In high risk situation a retreat system of working is normally preferred. However

the most important advantage gained by adopting a retreating system is lost if an attempt is made to ventilate the waste using a bleeder entry system.

Rate of Advance: Within any waste area adjacent to working place there will be air entering the waste, either by force of ventilating current or deliberately induced by a bleed action. In this area the rate of airflow can be critical. In practice, when a working face is operating normally, any individual piece of coal passes through the zone at a rate equal to the rate of advance of the working face. It is the time taken from entering or leaving the zone that is critical. If the time is excessive, the oxidation may occur to an unacceptable degree and a gob fire could result.

Crushing: It is significant in two types of locations; at pillar and rib edges and at worked out areas, where a pillar is subjected to crush, a situation can develop where leakage paths are created, lending both to the flow of air into the coal and, in some circumstances, through the solid to affect a more distant zone. Loose coal is usually present in worked out areas, and is produced either by pillar spalling or by roof collapses with associated sluggish ventilation. These areas are predominantly spontaneous combustion hazards.

Ventilating Pressure: The flow of air in mines is necessarily associated with pressure difference. This pressure difference is created by the mine flaws and natural ventilation, whilst the pressure distribution underground depends upon the resistance of the airways and the distribution of the quantity. Air will try to flow wherever there is a path and a pressure difference, and this means that air will flow along any break or crack in the strata that is open to a pressure difference. A high ventilating pressure differential factor appears to be an important factor contributing to spontaneous combustion. A number of heatings have developed after the ventilation system has been distributed or altered by installing a new mine fan or by introducing or withdrawing a secondary ventilation system.

Barometric pressure: It is generally agreed that air may find its way into a sealed-off area as a result of ccontinuous leakage resulting from a difference in pressure in the return and intake stoppings, or barometric changes or fluctuations in ventilating pressure resulting from the opening

of doors and the movement of cages and mine cars. Although leakage is very frequently responsible for starting fresh heating or the fanning up of old heating, the influence of barometric changes is believed by many to be of paramount importance. The emission of methane exposes more coal surface for oxidation and subsequent heating.

Leakage: To create the circumstances in which spontaneous combustion can occur, there must be a supply of oxygen and a situation where a build-up of heat is possible. This can be brought about by air leaks through fissures in solid coals and can result in a shallow-seated heating. This situation can occur where leakage paths exist at air crossings, in and around regulators and doors, and other similar locations where there is a high pressure gradient and tendency for air to attempt to flow through solid coal. The leakage through a stopping depends partly on the permeability of the stopping. It is not feasible to rely on making stoppings impermeable and if they were completely impermeable a dangerous pressure of firedamp could be seen accumulating. Rise of barometric pressure will doubtless tend to drive air through the stoppings, but in a gassy seam this will probably be prevented by the continuous evolution of firedamp behind the stoppings.

3.1 Spontaneous Heating Theories

Coal on introduction to air oxidizes. The oxidation methodology of coal is exceedingly exothermic in nature. The responses happening in beginning periods of unconstrained heating are normally physical in nature. The oxygen is adsorbed at the coal surface bringing about arrival of expansive measure of heating. Rate of heating discharge in oxidation of coal changes from 2.0 to 4.0 Cal for every mole of oxygen adsorbed. Beginning rate of oxygen adsorption is high which then slowly diminishes. The released heat generally gets scattered into environment and again it gathers and the results occurring change to chemical in nature (Banerjee, 1985).

Pyrite Theory: Heating of coal can be caused by iron pyrites only when present in considerable proportion and in freely powdered and dispersed state in the presence of moisture. The reaction of iron pyrites with oxygen and moisture is exothermic and yields

products of greater volume than the original pyrite thus opening more per area for oxygen. These equations shows that both oxygen and moisture, the two prime weathering agents, contribute to pyrite alteration and that sulphuric acid is formed as a by-product of the alteration. In comparison to dry coals in the presence of moisture, the reactivity of coal is formed to be double and if pyrite is in finely dispersed state it becomes 10 fold. It was seen that pyrite below 5% showed less or negligible effect.

$$2FeS_2 + 7O_2 + 16H_2O \rightarrow 2H_2SO_4 + 2FeSO_4.7H_2O + 316Kcal$$

$$FeS_2 + 15/4O_2 + 5/2H_2O \rightarrow FeO.OH + 2H_2SO_4$$

Bacterium Theory*:* This theory has its root in the occurrences of spontaneous heating in hay stacks and wood due to bacterial action. Coward conducted 6 observations between 1908 & 1927 and found distinct evidence showing that bacteria were capable of living on coal and in some cases such bacteria could cause a slight rise in temperature of the coal. Graham (1923-24) observed that ***s***terilized coal oxidized with the same pace as the unsterilized coal and concluded that oxidation mechanism did not include the activity of bacteria.

Phenol Theory*:* Different analysis have shown that phenolic hydroxyls and polyphenols oxidize faster than of many other groups. This theory is very interesting because it provides a method of determining susceptibility of coals to spontaneous heating.

Electro-chemical Theory*:* It provides auto-oxidation of coals as oxidation reduction phenomenon in micro-galvanic cells formed due to the coal components.

Coal-oxygen Complex Theory*:* Oxidation of coal is believed to be initiated at native radical sites. Formation of peroxyl radicals and hydro-peroxide is commonly thought to be the mechanism by which oxygen and moisture are initially incorporated into organic matter. These species may then react and rearrange or decompose to form a wide range of oxygen functionality in the matrix or gaseous products.

CHAPTER 4

EXPERIMENTAL INVESTIGATIONS

4. EXPERIMENTAL INVESTIGATIONS

In order to carry out this study, a number of coal samples were collected from different mines of all the major coalfields of the country. One sample from New South Wales, Australia; was also used in this study. The intrinsic properties of these samples were determined by proximate analysis and bomb calorimetry. The mineralogical composition was studied by XRD analysis. The spontaneous heating susceptibility indices were determined Differential Thermal Analysis (DTA) and Fourier Transform Infrared Spectroscopy (FTIR).

4.1 Sample Collection and Preparation

Sampling is the process of collecting the small portion of a whole such that consistence of that portion represents that of a whole. The samples were collected by using the channel sampling method (IS 436 Part I/Section I, 1964). The method consisted of cutting channels across the face of exposed ore and collecting the resulting chips, fragments and dust from each channel to make up a sample. First, a coal seam was chosen and the entire length upto a width of 10 cm of it demarcated by chalk was cut to a depth of 2.5cm. In selecting the place for sampling dirt bands exceeding have to be avoided.

4.1.2 Steps of channel sampling

The channel sampling consisted of the following steps:

Preparation the surface: Before cutting the surface, the exposure was cleaned to remove dust, shine, pre-oxidised coal and soluble salts. This may be done by washing the rock thoroughly with a hose or scrubbing with a stiff brush. It was done by chipping off the outer layer of the rock upto a depth of 10 cm along the band within which the sample was to be taken.

Demarcation of the channel: When the surface was cleaned, the next step was to mark out the location of the channel by inscribing two parallel lines on the ore body 12-15 cm apart using a chalk or paint.

Cutting the channel: Next, the channel was cut using a hand pick (prospectors pick). Usually a hammer and a moil are used to cut the grooves. The hammer weight should be 1.5 kg for single-handed work and 3.5 kg for double-handed work. Sufficient no. of moils should be kept so that work is not delayed owing to blunting of moils. However a pick is convenient for soft rock like coal and it cannot cope with hard ore bodies. In some mines, the sampling process is speeded by using a light air operated machine drill equipped with a pointed or chisel shaped bit or a coal drill in case of underground mines where more number of samples are needed to be taken in a single shift.

Collection the sample: A sheet of canvas was spread on the floor so as to catch the coal chips as they fall.

Labelling the sample: The collected sample was placed in a canvas sack along with an identifying label or tag. It was then brought to the surface.

For assaying the volume of sample has to be substantially reduced. This has to done such that the reduced volume retains the true composition of the original volume and for this it is necessary to comminute the samples so that no particle in the samples exceeds a certain maximum size. Samples can be comminuted mechanically but for size reduction on the spot particularly with channel sampling a hammer and an anvil may be used to break the large pieces in the samples. Usually steel, ring of 15 mm diameter with a handle is used to surround the sample and prevent flying particles when breaking should be on the sampling canvas an quartering floor so that pieces do not fly away and get lost. The sheet should be brushed clean by a whisk brush so as not lose any fines for reduction of samples coning and quartering process should be used until the desired size and volume of samples is obtained.

The sample from different coal fields were brought to the laboratory. The relatively big pieces were crushed to small pieces. Coning and quartering procedure was done to get a representative sample of the entire coal sample. This was then ground and screened (sieving) to a size of - 212μ (micron). Finally, the samples were kept in air-tight plastic bags for further analysis and to isolate them from external factors which might result in some change of its properties.

Fourty nine coal samples were collected by following the above procedure, and the list of samples has been presented in Table 4.1.

Table 4.1: List of Samples Collected

Sl. No.	Sample Name
1.	SCCL-1
2.	SCCL-2
3.	SCCL-3
4.	SCCL-4
5.	SCCL-5
6.	SCCL-6
7.	SCCL-7
8.	SECL-1
9.	SECL-2
10.	SECL-3
11.	SECL-4
12.	SECL-5
13.	SECL-6
14.	SECL-7
15.	CCL-1
16.	CCL-2
17.	ECL-1
18.	ECL-2
19.	ECL-3
20.	ECL-4
21.	ECL-5
22.	ECL-6
23.	NEC-1
24.	MCL-1
25.	MCL-2

Sl. No.	Sample Name
26.	MCL-3
27.	MCL-4
28.	MCL-5
29.	MCL-6
30.	MCL-7
31.	MCL-8
32.	MCL-9
33.	MCL-10
34.	MCL-11
35.	MCL-12
36.	MCL-13
37.	MCL-14
38.	MCL-15
39.	MCL-16
40.	MCL-17
41.	MCL-18
42.	MCL-19
43.	BCCL-1
44.	BCCL-2
45.	BCCL-3
46.	WCL-1
47.	NLC-1
48.	NCL-1
49.	NSW-1

4.2 Determination of Intrinsic Properties of Coal

Proximate analysis represents the percentage by weight of the Fixed Carbon, Volatiles, Ash, and Moisture Content in coal. Whereas the amounts of fixed carbon and volatile matter present in the coal directly contribute to the heating value of coal. Fixed carbon present in the coal acts as a main heat generator during heating process leads to burning. Content of high volatile matter indicates easy ignition of fuel. The content of ash is important in the design of the furnace grate, combustion volume, pollution control equipment and ash handling system of a furnace. These variables are presented in an air dried basis. This basis neglects the presence of moisture other than inherent moisture.

4.2.1 Proximate analysis (IS 1350 part I -1984)

Method used to analyse the proximate analysis was under IS 1350 part I -1984. The procedures were used to find the moisture %, volatile matter % and ash %.

4.2.1.1 Determination of Moisture Content

Coal, due to its nature, origin and occurrence, is always associated with some amount of moisture that is both physically and chemically bound. It is differentiated between inherent and external moisture. When a wet coal is exposed to atmosphere, the external moisture evaporates, but the dry coal still have some moisture, that can be removed only on heating above 100°C. External moisture is also known as accidental or free moisture, whereas inherent moisture is known as equilibrium or air-dried or hygroscopic moisture. The external moisture quantity depends on the mode of occurrence and coal handling, but the air-dried moisture is related to the inherent hygroscopic nature of the coal.

Experimental Procedure: 1g of finely powdered coal sample (-212μ) was weighed in a crucible of silica and was placed inside an electric hot air oven (Fig. 4.1) maintained at 108° +/- 2^0C. The crucible along with the sample of coal was kept in the oven for about 1.5 hours and was then taken out with a pair of tongues and hence cooled in a desiccator for about 15 minutes and then weighed. The loss in weight was termed as moisture content of the coal (on percentage basis).

$$\% \text{ Moisture} = \frac{Y - Z}{Y - X} * 100$$

Where,

X = weight of empty crucible, in g

Y = weight of crucible + coal sample before heating, in g

Z = weight of crucible + coal sample after heating, in g

Y – X = weight of coal sample, in g

Y- Z = weight of moisture, in g

Figure 4.1: Oven for Moisture Content Determination

4.2.1.2 Determination of Volatile Matter Content

The mass loss, corrected for moisture, that results when coal is heated in specified equipment under prescribed conditions, is termed as volatile matter. The lost material is composed of materials that form due to thermal decomposition of the different components of coal. Some of the constituents of coal which are termed as volatile matter are hydrogen, carbon monoxide, methane, other hydrocarbons, tar vapours, ammonia, some organic sulphur, oxygen containing compounds and some incombustible gases, such as carbon dioxide and water vapour.

Experimental Procedure: For the determination of volatile matter a special volatile matter silica crucible (38mm height, 25mm external diameter and 22mm internal diameter) was used in the experiment. The empty crucible was weighed. Approximately 1g of coal sample (-212 size) was weighed in the volatile matter crucible and it was placed inside a

muffle furnace maintained at 925°C with the lid covering the crucible. The heating was carried I the muffle furnace (Fig no. 4.2.2) out exactly for seven minutes, after which the crucible was removed, cooled in air, then in a desiccator and weighed again. The calculation was done as per the following.

$$\% \text{ Volatile Matter} = \frac{Y - Z}{Y - X} * 100 - M$$

Where,

X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, in g

Z = weight of crucible + coal sample after heating, in g

Y -X = weight of coal sample, in g

Y- Z = weight of volatile matter + moisture, in g

Figure 4.2: Muffle Furnace for Determination of Volatile Matter and Ash Content

4.2.1.3 Determination of Ash Content

Coal ash is reported the residue remaining after the process of combustion of coal under specified conditions. It usually does not occur as such in the coal, but is formed due to the changes of chemical constituents that take place in the mineral matter during the process of combustion. Ash and mineral matter of coal are therefore not identical.

There are two different types of ash forming materials in coal: extraneous mineral matters and inherent mineral matters. The extraneous mineral matter consists of materials such as calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum. The extraneous mineral matter owes its origin to i) the substances which got associated with the decaying vegetable material during its conversion to coal, which is difficult to remove by mechanical methods, and ii) rocks and dirt getting mixed up during mining and handling of coal. Inherent mineral matter represents the inorganic elements combined with organic components of coal. The origin of such materials is probably the plant materials from which the coal was formed. Ash from inherent mineral matter is insignificant as far as the total quantity of ash is concerned. But Indian coals suffer from the major disadvantage, that the mineral matter content is not only high, but of intimately associated type, due to its drift origin.

Experimental Procedure: First the empty crucible was cleaned in a muffle furnace by heating for about one hour at 800°C so that other mineral matter if presents get burnt. It was then taken out, cooled to room temperature and the weight is taken. Approximately 1gm of coal sample was weighed in the crucible and placed in a muffle furnace at 450°C for 30 minutes and then the temperature of the furnace was raised to 850°C for 1hour. The crucible was taken out and placed in a desiccator and weighed.

$$\% \text{ Ash} = \frac{Z - X}{Y - X} * 100$$

Where,

X = weight of empty crucible in grams

Y = weight of coal sample + crucible in grams (Before heating)

Z = weight of coal sample + crucible in grams (After heating)

Y – X = weight of coal sample, in g

Z - X = weight of ash, in g

4.2.1.4 Determination of Fixed Carbon (FC)

Fixed carbon (FC) is nothing but the mathematical remaining after determine successfully the moisture, volatile matter and ash that are presents in the coal sample. It is, thereof a measure of the combustible solid material in coal after the expulsion of volatile matter. Fixed carbon along with ash reports the approximate yield of coke from coal.

The fixed carbon value is thus determined by subtracting from the resultant summation of moisture, volatile matter and ash from 100 with all percentage.

FC= 100-(VM+M+A)

The moisture (M), volatile matter (VM), Ash and Fixed carbon content of coal determined by the above procedure is presented in Table 4.2

4.2.2 Determination of Calorific Value (IS 1350, Part- II- 1970)

The calorific value of coal, or the fuel content, is thus the amount of potential energy stored in coal that can be converted into actual heatingvalue. The process of determining the heat involved in the chemical reactions or physical changes as well as heat capacity is called calorimetry and the object used for determining the energy value or calorific value is known as calorimeter. The value generally varies with grades of coal or materials as different materials of different grades generates different quantity of heat for a given sample of mass.

A bomb calorimeter consists of a small crucible to carry the given sample, oxygen cylinder, a stainless steel bomb, water jacket, a stirrer, a thermometer, the insulating container (to prevent heat flow from the calorimeter to the surroundings) and ignition circuit connected to the bomb. By using stainless steel for the bomb, the reaction will occur with no volume change observed. It is a type of constant-volume calorimeter used in measuring the heat of combustion of a particular reaction. They withstand a large pressure within the calorimeter as the reaction is being measured. In this method electrical energy is used to ignite the sample of fuel; as the fuel is burning, it will heat up the surrounding air, which expands and escapes through a tube that leads the air out of the calorimeter. When the air is escaping through the copper tube it will also heat up the water outside the tube. The temperature of the water allows for calculating calorie content of the fuel.

Experimental Procedure: About 1gm of coal was measured in the digital balance and the reading given to the bomb calorimeter using start pre-weight button and stored the sample name followed by it. Coal was then placed in the small crucible provided and fixed in the arrangement provided in the cover of bomb. A piece of nichrome wire was cut and

attached to the two rods below the cover such that the wire is in contact with coal. The photographic view of the bomb calorimeter has been presented in Fig. 4.3.

Figure 4.3: Photographic view of Digital Bomb Calorimeter (Parr) and Auto filling of Oxygen in Bomb

The whole arrangement was carefully screwed over the bomb and oxygen line was attached over the valve on the cover. Oxygen fill button was pressed and oxygen was filled in the bomb. After this the bomb was carefully placed inside the water jacket. The bomb is to be placed so that is does not come in contact with the stirrer. The leads are attached to the two terminals provided on the cover.

The lid of machine is closed and the Start button is pressed. After sometimes the machine will ignite the coal and display the Gross Calorific Value (GCV) on screen. The GCV obtained by the above procedure for all the coal samples is also presented in Table 4.2.

Table 4.2: Results of Proximate Analysis and Gross Calorific Value

SL NO	SAMPLE NAME	MOISTURE (%)	VOLATILE MATTER (V.M%)	ASH (%)	FIXED CARBON (F.C %)	Gross Calorific Value (Kcal/Kg)
1	SCCL-1	5.50	26.00	18.00	50.50	5708.50
2	SCCL-2	4.50	32.00	12.00	51.50	6290.20
3	SCCL-3	5.97	35.49	26.49	32.05	5015.32
4	SCCL-4	2.31	26.34	29.72	41.63	5425.22
5	SCCL-5	4.29	11.99	35.16	48.56	4624.71

6	SCCL-6	2.78	16.93	33.21	47.08	5028.17
7	SCCL-7	4.98	18.78	20.01	56.23	5950.77
8	SECL-1	5.00	26.50	17.00	51.50	5931.04
9	SECL-2	5.00	32.33	34.00	28.67	4316.27
10	SECL-3	7.67	29.83	18.88	43.62	7160.23
11	SECL-4	4.85	16.9	21.85	56.40	5796.44
12	SECL-5	9.96	12.60	30.39	47.05	4248.30
13	SECL-6	2.93	12.04	30.88	54.15	5225.73
14	SECL-7	7.67	10.80	30.92	50.61	4531.82
15	CCL-1	5.05	22.49	48.51	23.95	3156.57
16	CCL-2	10.00	32.27	18.00	39.73	5119.78
17	ECL-1	1.00	21.89	34.33	57.22	5337.35
18	ECL-2	1.00	24.62	26.50	47.88	6295.61
19	ECL-3	1.00	22.54	34.16	42.30	5426.97
20	ECL-4	1.00	22.11	34.33	42.56	5121.95
21	ECL-5	8.43	24.43	9.60	57.54	7315.23
22	ECL-6	1.80	36.13	14.27	47.80	3481.05
23	NEC-1	1.50	43.03	5.37	50.10	8075.08
24	MCL-1	6.50	32.00	23.50	38.00	4782.11
25	MCL-2	2.98	34.17	12.00	50.85	6691.48
26	MCL-3	11.13	25.19	38.46	25.22	3463.14
27	MCL-4	14.29	31.25	16.27	38.19	5156.32
28	MCL-5	7.25	23.90	40.50	28.35	3512.81
29	MCL-6	8.97	29.49	22.88	38.66	4987.39
30	MCL-7	10.02	26.06	31.57	32.35	3999.07
31	MCL-8	11.32	29.81	21.80	37.07	4896.30
32	MCL-9	9.12	33.51	8.37	48.91	6377.20
33	MCL-10	8.72	25.14	13.75	52.39	6147.50
34	MCL-11	6.74	26.14	34.89	32.23	3802.35
35	MCL-12	7.64	22.13	37.02	33.21	4112.82
36	MCL-13	5.00	24.01	32.40	38.59	4027.36
37	MCL-14	4.88	24.17	38.87	32.08	3800.69
38	MCL-15	6.31	28.39	32.42	32.88	2366.66

39	MCL 16	14.5	31.97	12.35	41.18	5285.92
40	MCL 17	6.65	30.31	22.53	40.51	5470.33
41	MCL 18	6.94	28.48	28.22	36.36	4892.34
42	MCL-19	6.7	11.5	40.9	40.90	3733.33
43	BCCL-1	1.90	33.08	16.00	49.02	6858.61
44	BCCL-2	0.60	22.32	10.73	66.35	6697.94
45	BCCL-3	1.00	17.36	15.73	65.91	7177.43
46	WCL-1	14.39	29.31	12.76	43.54	5216.94
47	NLC-1	37.30	32.32	3.40	26.98	5017.34
48	NCL-1	9.68	29.80	18.12	42.40	5684.60
49	NSW-1	2.05	20.85	9.03	68.07	6052.67

4.2.3 MINERALOGICAL STUDY (XRD STUDIES)

One important factor that affects coal reactivity and hence spontaneous heating propensity is the mineral matter present in coal (Beamish and Arisoy, 2008). However, little is known about mineral matter effects on coal self-heating. In this work an attempt has been to identify the minerals present in coal by XRD-analysis. The collected coal samples were crushed to -200 mesh size. The sample in powder form was then put into the glass mould for putting it in the Multipurpose X-Ray Diffraction System (Rigaku Japan/Ultima-IV) as shown in Figure 4.4(a). A copper target is used in this machine of wavelength 1.54 Å (Fig. 4.4(b)). The XRD plot obtained was then analysed with the help of X-Pert HighScore Plus Software. The specification of the XRD system has been presented in Table 4.3.

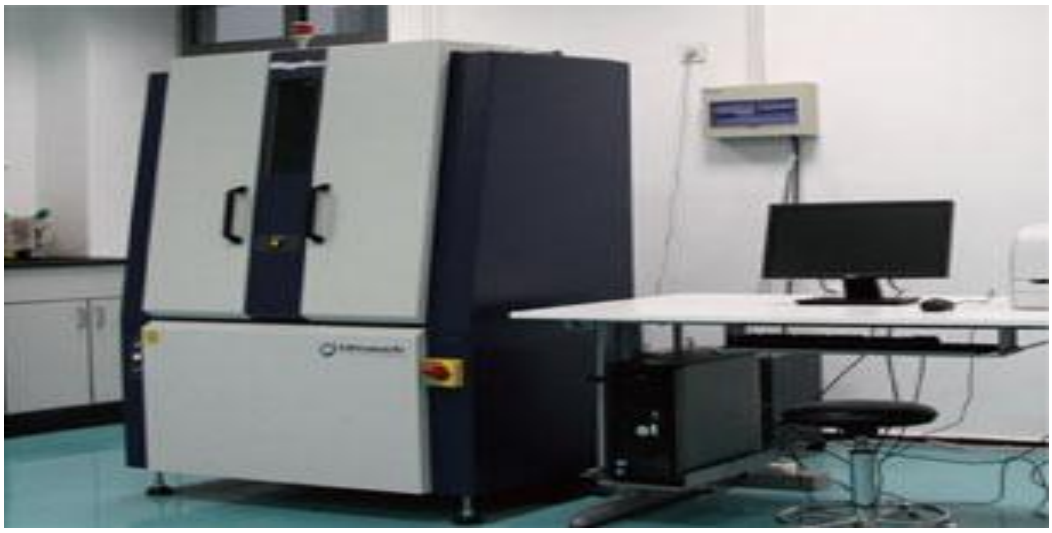


Figure 4.4(a): X-Ray Diffraction System (Rigaku Japan/Ultima-IV)

Figure 4.4(b): Sample holder and Copper target in XRD system

Table 4.3: Ultima IV X-Ray Diffraction System Specifications

X-ray generator	**Maximum rated output**	**3 Kw**
	Rated tube voltage	20 - 60 Kv
	Rated tube current	2 - 60 mA
	Target	Cu (others: optional)
	Focus size	0.4 x 12 mm (others: optional)
Goniometer	Scanning mode	θs/θd coupled or θs, θd independent
	Goniometer radius	285 mm
	2θ measuring range	-3 to 162° (maximum)
	Minimum step size	0.0001°
Optics	Divergence slit	Fixed or automatic variable
	Scattering slit	Fixed or automatic variable
	Receiving slit	Fixed or automatic variable
	Optics alignment	Automatic alignment of tube height, goniometer, optics and detector
	Monochromator	Dual position graphite diffracted beam monochromator for Cu (others: optional)
Detector	Detector	Scintillation counter (others: optional)
Dimensions	H x W x D	1600 x 1100 x 800 mm
	Sample height	1050 mm

Mineralogy analysis of some sample with the help of X-Pert HighScore Plus Software has been presented in Fig.4.5 and 4.6 respectively.

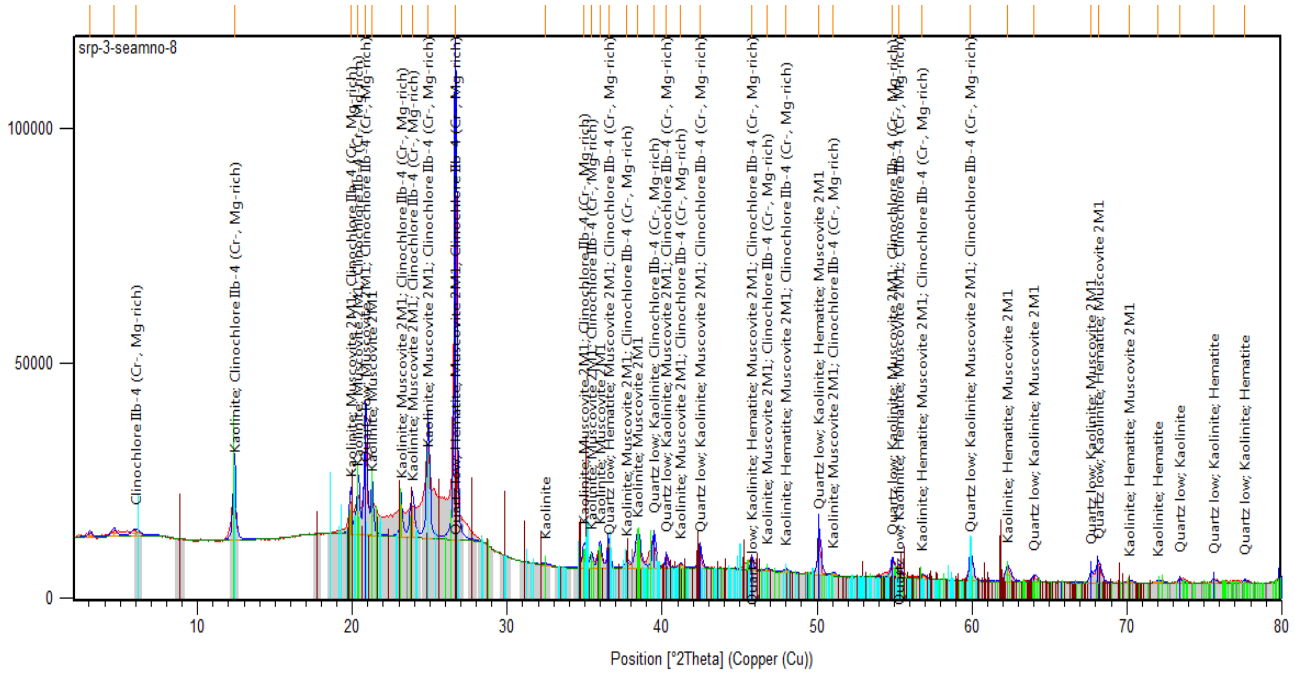


Figure 4.5: Peaks of Mineral in SCCL-1 Coal Sample

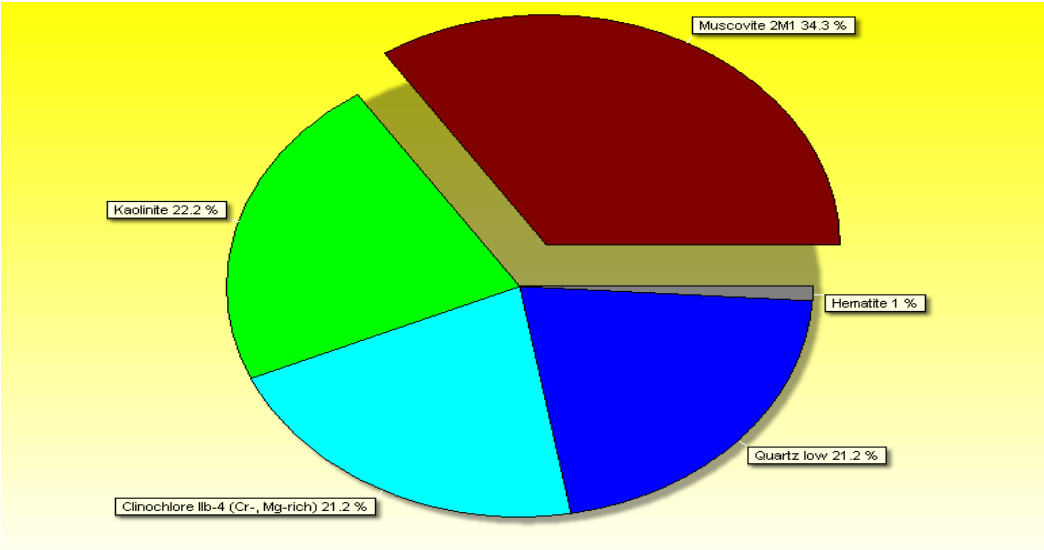


Figure 4.6: Quantification of Mineral of SCCL-1 Coal Sample

The mineralogy study of 49 coal sample has been presented in the Table 4.4.

Table 4.4: Mineralogy Studies of Coal Samples

SL NO	SAMPLE NAME	Calcite		Clinochlore		Gypsum		Hematite		Jarosite		Kaolinite		Muscovite		Orthoclase		Quartz		Sulfer	
1	SCCL-1	N	0	Y	21.2%	N	0	Y	1%	N	0	Y	22.2%	Y	34.3%	N	0	Y	21.2%	N	0
2	SCCL-2	N	0	Y	32.0%	N	0	Y	2%	N	0	Y	44.0%	N	0	N	0	Y	22%	N	0
3	SCCL-3	N	0	Y	9.9%	N	0	N	0	N	0	Y	17.8%	Y	24.8%	Y	21.8%	Y	25.7%	N	0
4	SCCL-4	N	0	N	0	N	0	N	0	Y	11%	Y	71%	N	0	N	0	Y	18%	N	0
5	SCCL-5	N	0	Y	2%	N	0	N	0	N	0	Y	32.3%	Y	23.2%	Y	15.2%	Y	12.1%	Y	15.2%
6	SCCL-6	N	0	Y	2%	N	0	Y	1%	N	0	N	0	Y	38%	Y	18%	Y	26%	Y	15%
7	SCCL-7	Y	5%	Y	23.8%	N	0	Y	3%	N	0	Y	29.7%	N	0	N	0	Y	38.6%	N	0
8	SECL-1	N	0	N	0	N	0	N	0	Y	9%	Y	63.0%	N	0	N	0	Y	28.0%	N	0
9	SECL-2	Y	3%	Y	15.2%	N	0	Y	1%	N	0	Y	19.2%	Y	39.4%	Y	4%	Y	18.2%	N	0
10	SECL-3	N	0	Y	9%	N	0	Y	1%	N	0	Y	21%	Y	20%	Y	7%	Y	33%	Y	9%
11	SECL-4	N	0	Y	12.9%	N	0	Y	1%	N	0	Y	20.8%	Y	25.7%	Y	5%	Y	16.8%	Y	17.8%
12	SECL-5	N	0	N	0	N	0	N	0	N	0	N	0	Y	71%	N	0	Y	29%	N	0
13	SECL-6	N	0	N	0	N	0	Y	2%	N	0	Y	28.7%	Y	5%	Y	5%	Y	59.4%	N	0
14	SECL-7	N	0	Y	19.8%	N	0	N	0	N	0	Y	31.7%	Y	4%	N	0%	Y	22.8%	Y	21.8%
15	CCL-1	N	0	N	0	N	0	N	0	N	0	Y	65%	Y	3%	N	0	Y	32%	N	0
16	CCL-2	N	0	N	0	N	0	N	0	N	0	Y	42%	Y	12%	Y	16%	Y	30%	N	0
17	ECL-1	N	0	N	0	N	0	N	0	N	0	Y	19%	Y	50%	Y	7%	Y	24%	N	0

18	ECL-2	N	0	N	0	N	0	Y	3%	N	0	Y	37%	Y	3%	Y	21%	Y	36%	N	0
19	ECL-3	N	0	N	0	N	0	Y	2%	N	0	Y	24%	Y	26.4%	Y	18.6%	Y	29%	N	0
20	ECL-4	N	0	N	0	N	0	Y	3%	N	0	Y	31.3%	Y	6.1%	Y	17.2%	Y	42.4%	N	0
21	ECL-5	N	0	N	0	N	0	Y	3%	N	0	Y	21.2%	Y	44.4%	N	0	Y	31.3%	N	0
22	ECL-6	N	0	N	0	N	0	N	0	N	0	Y	21.2	Y	42.4%	Y	4%	Y	32.4%	N	0
23	NEC-1	N	0	Y	15%	N	0	Y	3%	N	0	Y	13%	N	0	N	0	Y	35%	Y	34%
24	MCL-1	N	0	Y	13.9%	N	0	N	0	N	0	Y	20.8%	Y	36.7%	Y	6.9%	Y	21.8%	N	0
25	MCL-2	Y	15.8%	N	0	N	0	N	0	N	0	Y	50.5%	N	0	N	0	Y	33.7%	N	0
26	MCL-3	N	0	N	0	N	0	N	0	N	0	Y	17.8%	Y	36.6%	Y	5.9%	Y	22.8%	Y	16.9
27	MCL-4	N	0	N	0	N	0	N	0	N	0	Y	24.8%	Y	4%	Y	13.9%	Y	26.7%	Y	30.7%
28	MCL-5	N	0	Y	2.7%	N	0	N	0	N	0	Y	16%	Y	30%	Y	12.3%	Y	14%	Y	26%
29	MCL-6	N	0	Y	2.9%	N	0	Y	1%	N	0	Y	15%	Y	38%	N	0	Y	12%	Y	25%
30	MCL-7	Y	19%	N	0	Y	1%	Y	1%	Y	1%	Y	27%	Y	30%	N	0	Y	21%	N	0
31	MCL-8	N	0	Y	2%	N	0	N	0	N	0	Y	51%	Y	4%	Y	19%	Y	24%	N	0
32	MCL-9	N	0	Y	11%	N	0	N	0	N	0	Y	22%	Y	40%	N	0	Y	12	Y	15%
33	MCL-10	N	0	Y	23.8%	N	0	N	0	N	0	Y	20.8%	Y	16.8%	Y	9.9%	Y	7.9%	Y	16.9
34	MCL-11	N	0	N	0	N	0	N	0	N	0	Y	31%	Y	18%	Y	11%	Y	27%	Y	13%
35	MCL-12	N	0	N	0	N	0	Y	3%	N	0	Y	42%	Y	10%	N	0	Y	45%	N	0
36	MCL-13	N	0	Y	8.1%	N	0	Y	1%	N	0	Y	18.1%	Y	19.2%	Y	7.1%	Y	17.1%	Y	28.4%
37	MCL-14	N	0	N	0	N	0	Y	2%	N	0	Y	23%	Y	57%	N	0	Y	18%	N	0

38	MCL-15	N	0	N	0	N	0	N	0	N	0	Y	62%	Y	3%	Y	9%	Y	26%	N	0
39	MCL-16	N	0	N	24%	Y	0	Y	1%	N	0	Y	31%	Y	28%	N	0	Y	8%	Y	8%
40	MCL-17	N	0	Y	12.9%	N	0	N	0	N	0	Y	14.9%	Y	16.8%	Y	25.7%	Y	8.9%	Y	20.8%
41	MCL-18	N	0	Y	14%	N	0	Y	1%	N	0	Y	18%	Y	14%	Y	22%	Y	7%	Y	24%
42	MCL-19	N	0	N	0	N	0	Y	1%	N	0	Y	44.4%	N	0	Y	11.1%	Y	31.3%	Y	12.1
43	BCCL-1	N	0	N	0	N	0	Y	64%	N	0	Y	20%	N	0	N	0	Y	16%	N	0
44	BCCL-2	N	0	N	0	N	0	N	100%	N	0	N	0	N	0	N	0	N	0	N	0
45	BCCL-3	N	0	N	0	N	0	N	57%	N	0	Y	24.2	N	0	N	0	Y	18.8	N	0
46	WCL-1	N	0	N	0	N	0	N	0	N	0	Y	29.7%	Y	5.9%	Y	30.7%	Y	33.7%	N	0
47	NLC-1	N	0	N	0	N	0	N	0	N	0	Y	32%	N	0	Y	54%	Y	7%	Y	7%
48	NCL-1	N	O	Y	2.8%	N	0	Y	1%	N	0	Y	33%	Y	12%	Y	21%	Y	18.2%	Y	12%
49	NSW-1	N	0	N	0	N	0	N	0	N	0	Y	37%	Y	24%	Y	15%	Y	16%	Y	8%

4.3 Determination of Susceptibility Indices

4.3.1 Differential Thermal Analysis

Thermal analysis is the technique by which physical and chemical changes of a substance are measured as a function of temperature as the substance is subjected to a controlled heating rate. Differential thermal analysis is a technique in which the temperature of the substance under investigation is compared with the temperature of a thermally inert material such as alpha-alumina and is recorded with furnace temperature as the substance is heated or cooled at a predetermined uniform rate. If a sample undergoes no thermal change and the temperature difference is zero, then a base line parallel to recording instrument is given by the recording instrument. If the sample undergoes a thermal decomposition reaction with loss or gain of heat then the temperature gradient with respect to inert material will change producing a positive or negative ΔT depending on whether the transformation taking place in the sample is endothermic or exothermic. DTA is a quick method which does not require complicated instruments. With modern DTAs, the measurements can be programmed so that the analysis is not very time-consuming. The curve recorded with ΔT plotted on the ordinate and temperature or time on the abscissa is called the DTA curve or thermogram.

The schematic diagram of a DTA apparatus has been presented in Figure 4.7. The sample holder assembly consists of a thermocouple each for the sample and reference, surrounded by a block to ensure an even heat distribution. The sample is contained in a small crucible designed with an indentation on the base to ensure a snug fit over the thermocouple bead. The crucible may be made of materials such as pyrex, silica, nickel or platinum, depending on the temperature and nature of the tests involved.

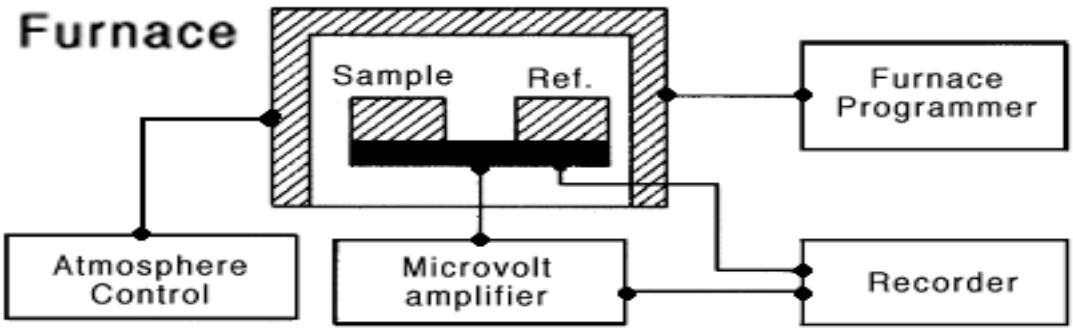
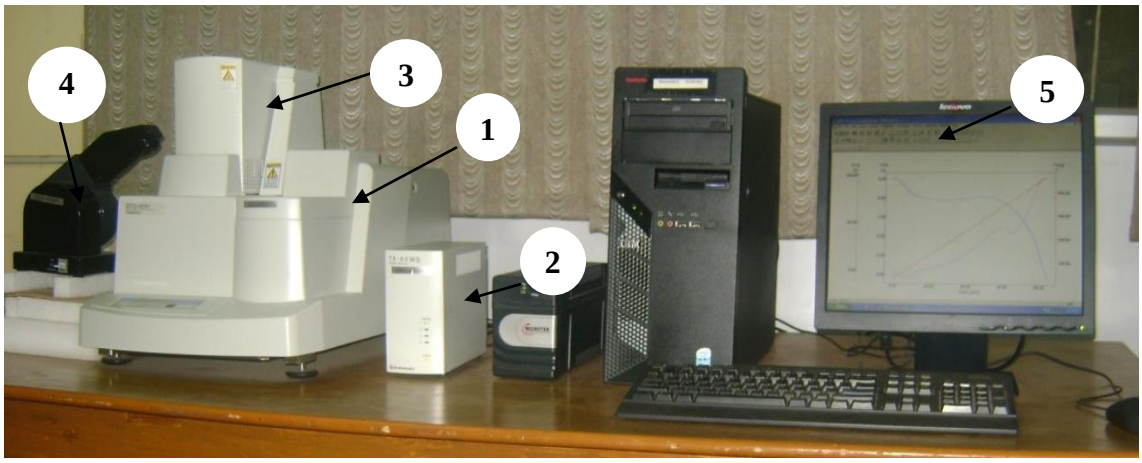


Figure 4.7: Schematic diagram of a typical DTA apparatus

Experimental Procedure: In the present study a differential thermal analyser DTA 60/60H (Schimadzu) was used (Fig. 4.8). Standardized parameters suggested by Banerjee and Chakravorty (1967) were followed while performing the experiments. DTA thermograms were obtained by maintaining a heating rate of 5^0C per min and alumina powder was taken as the reference material. Thermogarms for all the samples were obtained by following this procedure and have been presented in Appendix -I.



1) Differential Thermal Analyser (DTA- 60H)
2) TA – 60 WS Collection Monitor
3) Tubular Furnace
4) Blower
5) Computer for Data Processing

Figure 4.8: Experimental Set up of Differential Thermal Analysis Apparatus

It has been shown by Banerjee and Chakravorty (1967) that a thermogram of coal can be divided into three segments or stages. In the initial stage of heating (stage I), the endothermic reaction predominates, probably due to the release of inherent moisture in coal. In the second stage (stage II), the exothermic reaction becomes significant, but the rate of heat release is not steady all through, as it changes with temperature. A steep rise in heat evolution is observed in the third stage (stage III). The same observation has been made in this study.

The rate of temperature rise in stage II is cited by different researchers (Banerjee and Chakravorty,1967; Gouws and Wade,1989, Sahu et al, 2005) as being less for coals with less susceptibility to spontaneous heating. The exothermicity in stage III is not regarded as a reliable indicator of the self heating risk, because it may be equally high for low rank coals. However, the temperature of transition or characteristic temperature or onset

temperature is considered to be significant. It is considered that in lower temperatures, the coal is more susceptible towards spontaneous heating.

Therefore, all the thermograms were analyzed for the following details:

Onset temperature or characteristic temperature

The onset temperature or characteristic temperature was determined by the following procedure:

- A tangent was drawn at the inflexion point of the endothermic region and another tangent was drawn at the rising portion of the curve of stage III.
- The intersection between the two tangents gives the characteristic temperature.
- The depiction of characteristic or onset temperature (T_c) from DTA thermogram has also been presented in Fig.4.9.

Slopes of stage II of the thermogram

In the thermograms obtained from the experiments, linear stage II exothermicity is not observed. In view of the disjointed nature of stage II slopes it was further divided into two different regions, viz. stage IIA and stage IIB. The following three parameters of stage II were determined for further analysis.

- Average slope of stage IIA.
- Average slope of stage IIB.
- Overall slope of stage II.

The characteristic or onset temperature (Tc) and the slopes of stage IIA, IIB and overall slope of stage II of DTA thermograms were obtained by using the TA-60 software provided with the equipment and the results have been presented in Table 4.5.

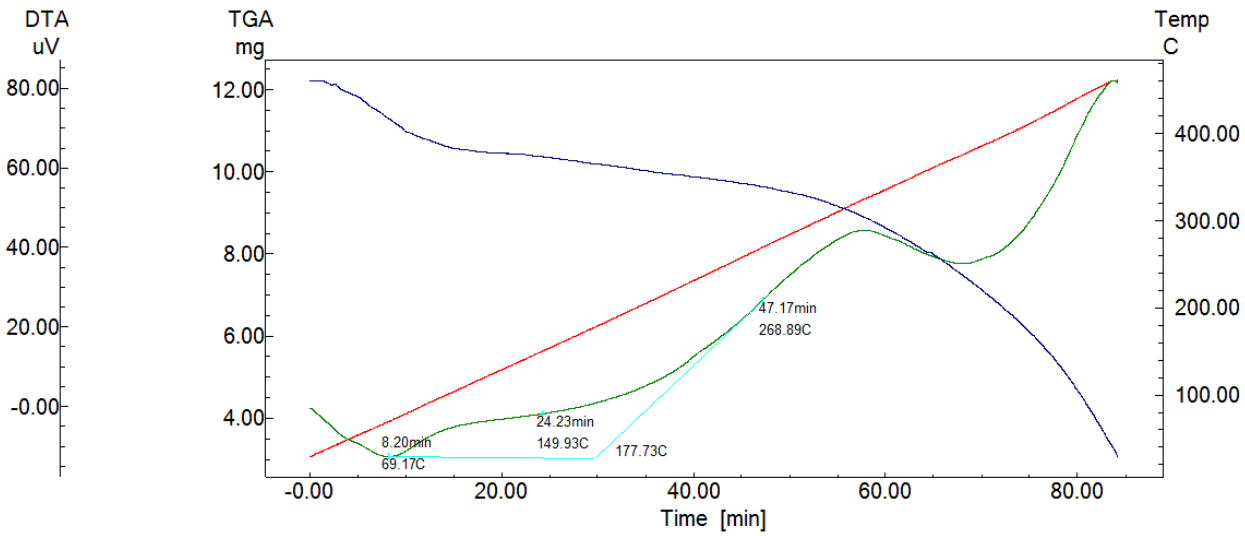


Figure 4.9: Determination of onset temperature from DTA Thermogram for SCCL-1 Coal Sample

Table 4.5: Transition Temperature and Slopes obtained from DTA Thermograms

Sl. NO.	SAMPLE NAME	Transition Temperature (^{0}C)	Slope		
			IIA	IIB	II
1.	SCCL-1	177.73	0.1329	0.2360	0.1611
2.	SCCL-2	197.82	0.1429	0.3149	0.2063
3.	SCCL-3	182.20	0.1222	0.3516	0.1723
4.	SCCL-4	168.02	0.0720	0.1098	0.0772
5.	SCCL-5	158.95	0.1367	0.2419	0.1581
6.	SCCL-6	151.70	0.1230	0.1843	0.1319
7.	SCCL-7	115.39	0.1844	0.1473	0.1433
8.	SECL-1	175.15	0.1274	0.3345	0.1747
9.	SECL-2	188.42	0.0974	0.3183	0.1567
10.	SECL-3	172.55	0.1051	0.1646	0.1047
11.	SECL-4	159.83	0.0786	0.0938	0.1647
12.	SECL-5	168.15	0.0799	0.1517	0.0900
13.	SECL-6	167.53	0.0870	0.1416	0.0947
14.	SECL-7	167.18	0.0635	0.1329	0.0784
15.	CCL-1	192.00	0.0812	0.2904	0.1274
16.	CCL-2	180.03	0.1129	0.2277	0.1291
17.	ECL-1	234.54	0.0103	0.2556	0.0522

18.	ECL-2	230.77	0.0213	0.3321	0.0744
19.	ECL-3	233.33	0.0053	0.1784	0.0529
20.	ECL-4	235.41	0.0122	0.2837	0.0546
21.	ECL-5	238.44	0.0045	0.1275	0.0332
22.	ECL-6	178.16	0.0901	0.1466	0.0922
23.	NEC-1	178.44	0.0472	0.2831	0.1132
24.	MCL-1	190.20	0.1837	0.4113	0.2298
25.	MCL-2	174.36	0.0275	0.1849	0.0527
26.	MCL-3	198.00	0.0712	0.2481	0.1103
27.	MCL-4	152.74	0.1380	0.1551	0.1194
28.	MCL-5	176.67	0.0755	0.1361	0.0800
29.	MCL-6	198.49	0.1053	0.2281	0.1331
30.	MCL-7	182.90	0.0905	0.1825	0.1083
31.	MCL-8	168.25	0.0981	0.1614	0.1004
32.	MCL-9	167.98	0.0906	0.1574	0.0963
33.	MCL-10	164.58	0.0927	0.1740	0.1038
34.	MCL-11	193.29	0.1027	0.2349	0.1327
35.	MCL-12	189.46	0.0984	0.2469	0.1300
36.	MCL-13	195.15	0.0681	0.1354	0.0065
37.	MCL-14	185.08	0.0806	0.1878	0.0999
38.	MCL-15	187.56	0.0574	0.1321	0.0676
39.	MCL-16	178.32	0.0773	0.2192	0.1088
40.	MCL-17	158.47	0.0506	0.2083	0.0782
41.	MCL-18	190.02	0.0690	0.1795	0.0957
42.	MCL-19	178.77	0.0657	0.1770	0.0950
43.	BCCL-1	206.48	0.0327	0.2430	0.1044
44.	BCCL-2	213.41	0.0163	0.1354	0.0540
45.	BCCL-3	235.12	0.0021	0.1339	0.0301
46.	WCL-1	172.55	0.1651	0.2272	0.1575
47.	NLC-1	192.47	0.0573	0.2000	0.1001
48.	NCL-1	193.54	0.0763	0.1569	0.0819
49.	NSW-1	216.22	0.0138	0.0993	0.0334

4.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR technique is normally used to get an infrared absorption and transmission spectrum of a sample that may be solid, liquid and gas. In FTIR the sample is analysed for a long range of wavelength at once. The result is then transformed to actual spectrum using Fourier transform (a mathematical process).

When a sample is exposed to infrared radiation containing a wide range spectrum of frequencies, the chemicals present inside the sample will absorb only certain wavelength and transmits the rest.

The light from a broadband source shines into a Michelson interferometer which is a configuration of mirrors controlled by a set of motors. The motors move the mirrors in such a way that a particular wavelength of light gets blocked and then transmitted and the blocked periodically. This is repeated with every wavelength so that each time a different spectrum is obtained. The detector receives and record the intensities of each wavelength reaching it. This raw data is the fed into the computer which uses a n algorithm known as Fourier transform to convert the raw data “Interferogram” into the light absorption or transmission spectrum for each wavelength.

Figure 4.10: Photographic view of FTIR Machine

Experimental Procedure: The coal samples used were air dried at 40^0C. The coal sample is sieved with 200 size mesh. KBr is used to make pellets and the range of spectra was 400 – 4000 cm^{-1} with a resolution of 4 cm^{-1}. For the determination of FT-IR spectroscopy of coal sample, the sample is mixed with potassium bromide (KBr) in the ratio 1:10. After thorough mixing the homogenous mixture was pressed into pellets using a pressing machine applying a pressure of 10 tonnes (Fig. 4.11). To normalize the effect of KBr during the scanning of coal samples, a background pellet of KBr was scanned before the scanning of coal samples to create a baseline for the given coal samples. All pellets were kept in a dessicator for 2 hours prior to testing. The Transmittance (T) plot for all coal samples obtained from FTIR is presented in Appendix – II. A sample FTIR spectra is presented in Figure 4.12.

Figure 4.11: Hydraulic Press

Determination of Carbon aliphaticity and aromaticity:

Absorbance, A, is represents the quantity of light absorbed from the initial incoming light, which is the base 10 logarithm of the reciprocal of transmittance.

$$A = \log_{10} \frac{1}{T}$$

Where, A is absorbance and T is transmittance in fraction.

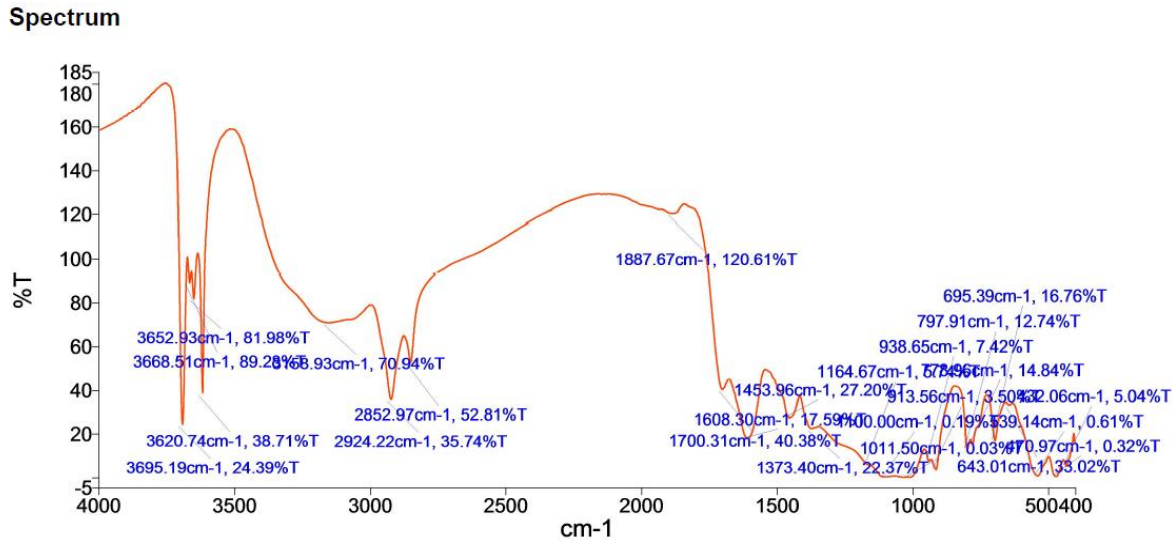


Fig. 4.12: FTIR Transmittance spectra for MCL – 3 Coal Sample

Beer-Lambert Law

The Beer-Lambert law represents the linear relationship between the concentration of absorbing species and the absorbance in the sample. The general law is:

$$A = a \times b \times c$$

Where,

a = the wavelength dependent absorvity coefficient

b = the path length

c = the concentration of species

Carbon aliphaticity is defined as:

$$F_{al} = \frac{\text{Absorbance at 2925 cm}-}{\text{Absorbance at 1600 cm}-}$$

And carbon aromaticity is termed as

$$Far = \text{————————————}$$

The absorbance around 2920 cm-1 represents C-H stretching frequency of CH_2 and CH_3 showing presence of aliphatic compounds, while C=C stretching frequency is observed around 1600 cm-1 representing aromatic species of different raw coals. The value of FAr-

IR changes with variation in the values of either absorbance of the stretching frequency around 1600 cm-1 or 2920 cm-1. The increase in value of FAr-IR could be attributed either due to an increase in the aromatic content or decrease in the aliphatic content of the coal. Therefore, it follows that even when the absorbance at aliphatic absorption frequency (aliphatic content) alone decreases without a significant change in aromatic content, the value FAr-IR appears to be increasing, while in reality aliphatic content is decreasing without any changes in aromatic content. Thus the value FAr-IR always represents the structural changes in coal only in terms of aromatic content of the coal irrespective of whether the actual changes are in aromatic or aliphatic contents and many times it fails to directly indicate the actual changes in the aliphatic content of the coal.

2925 cm^{-1} provides the information about the aliphatic bonds C-H whereas the 1600 cm- represents the aromatic bonds or conjugate bonds. Higher the aliphaticity, higher is the tendency of coals towards spontaneous heating. The aliphaticity results of all coal samples are given in Table 4.6.

Table 4.6: Aliphaticity of Coal Samples

Sample No	Sample Name	Aliphaticity
1	SCCL-1	1.42
2	SCCL-2	1.06
3	SCCL-3	0.97
4	SCCL-4	1.06
5	SCCL-5	0.94
6	SCCL-6	0.93
7	SCCL-7	2.37
8	SECL-1	1.05
9	SECL-2	1.10
10	SECL-3	1.11
11	SECL-4	1.11
12	SECL-5	1.10
13	SECL-6	1.13

Sample No	Sample Name	Aliphaticity
32	MCL-9	1.00
33	MCL-10	1.15
34	MCL-11	1.42
35	MCL-12	1.11
36	MCL-13	1.27
37	MCL-14	1.23
38	MCL-15	1.43
39	MCL-16	1.20
40	MCL-17	1.21
41	MCL-18	1.11
42	MCL-19	2.12
43	BCCL-1	0.95
44	BCCL-2	0.94
45	BCCL-3	0.95
46	WCL-1	1.21
47	NLC-1	2.35
48	NCL-1	2.33
49	NSW-1	1.09

Sample No	Sample Name	Aliphaticity
14	SECL-7	1.09
15	CCL-1	1.17
16	CCL-2	1.15
17	ECL-1	0.73
18	ECL-2	2.01
19	ECL-3	0.80
20	ECL-4	0.89
21	ECL-5	0.74
22	ECL-6	1.03
23	NEC-1	0.98
24	MCL-1	1.09
25	MCL-2	1.03
26	MCL-3	1.22
27	MCL-4	1.14
28	MCL-5	1.14
29	MCL-6	1.33
30	MCL-7	1.41
31	MCL-8	1.17

CHAPTER 5

CORRELATION STUDY

5. CORRELATION STUDY

Correlation study was carried out to determine the relationship between the intrinsic properties of coal and aliphaticity with the susceptibility indices. The intrinsic parameters determined by proximate analysis and bomb calorimetry were taken as independent variables; whereas Tc, slopes of stage IIA, IIB and overall slope of stage II obtained from DTA thermogarms, and carbon aliphaticity obtained from FTIR studyconsidered as depended variables. Multi-variable correlation was also done to get the combined effect of the proximate constituents with the susceptibility indices and the effect of aliphaticity with proximate constituents. The results of the correlation study has been presented in Table 5.1. The correlation plots for the same has been presented in Figure 5.1 to have been along with the different plots have been presented below:

Table 5.1: Correlation between Intrinsic Properties and Susceptibility Indices

Sl. No.	Independent variable	DTA				FTIR
		Tc	IIA	IIB	II	Aliphaticity
1.	M	0.62	0.64	0.08	0.46	0.48
2.	VM	0.34	0.58	0.36	0.58	0.08
3.	A	0.19	0.15	0.13	0.23	0.43
4.	FC	0.39	0.61	0.38	0.74	0.70
5.	GCV	0.22	0.88	0.24	0.32	0.82

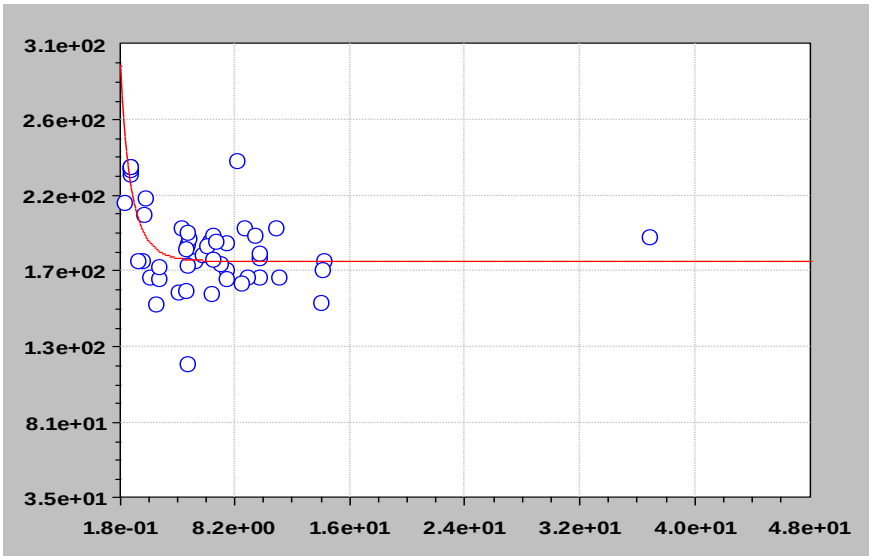


Figure 5.1: Correlation plot between Tc and Moisture content

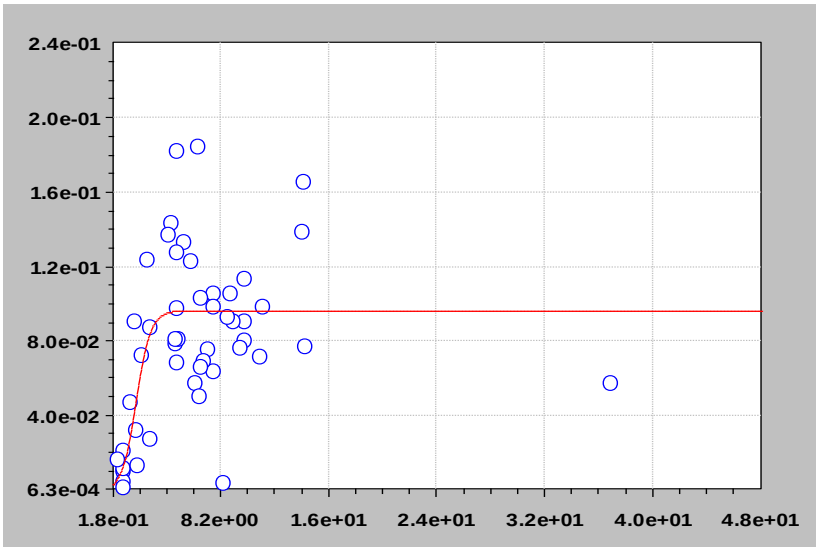


Figure 5.2: Correlation plot between IIA Slope and Moisture content

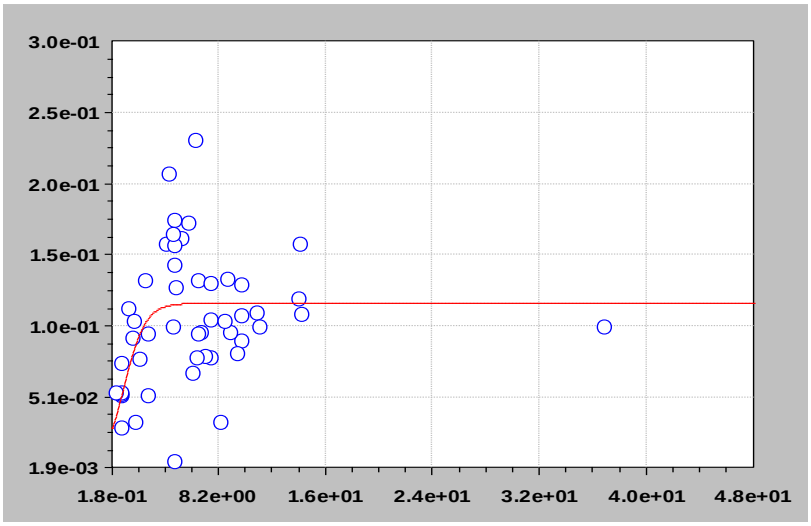


Figure 5.3: Correlation plot between II Slope and Moisture content

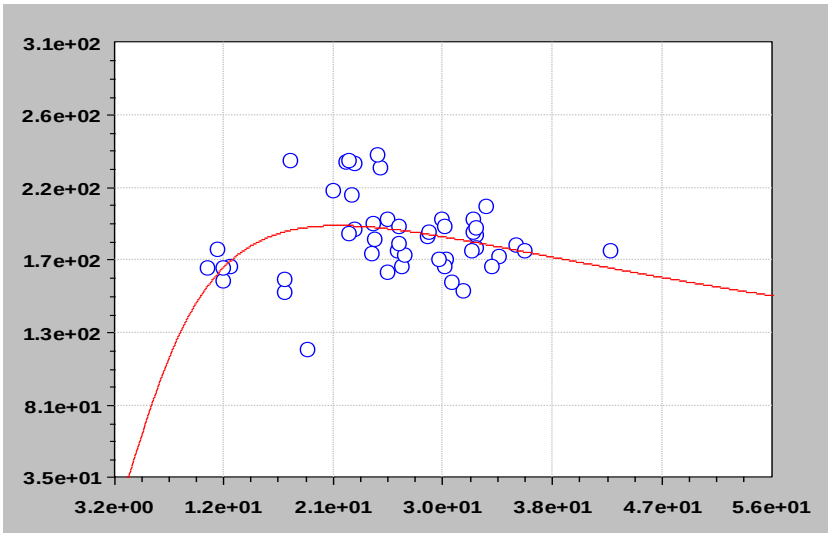


Figure 5.4: Correlation plot between Tc and Volatile matter Content

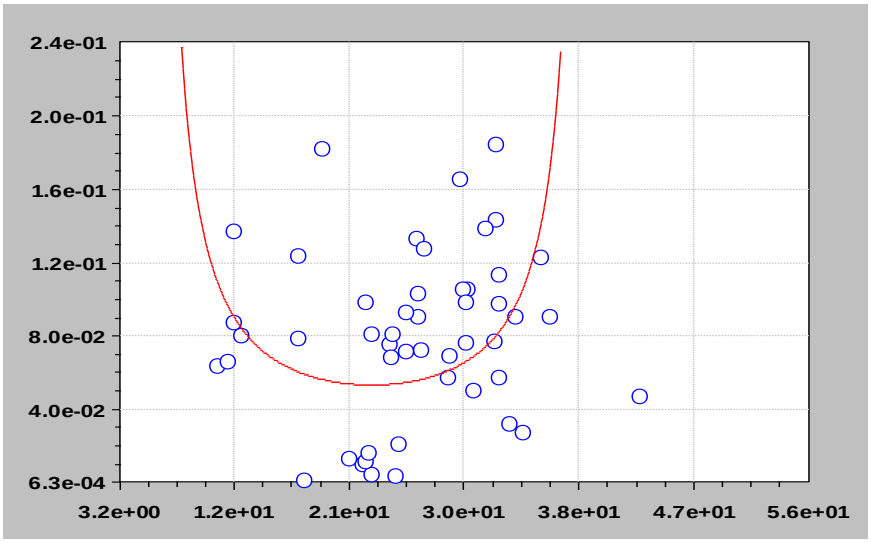


Figure 5.5: Correlation plot between II A Slope and Volatile Matter Content

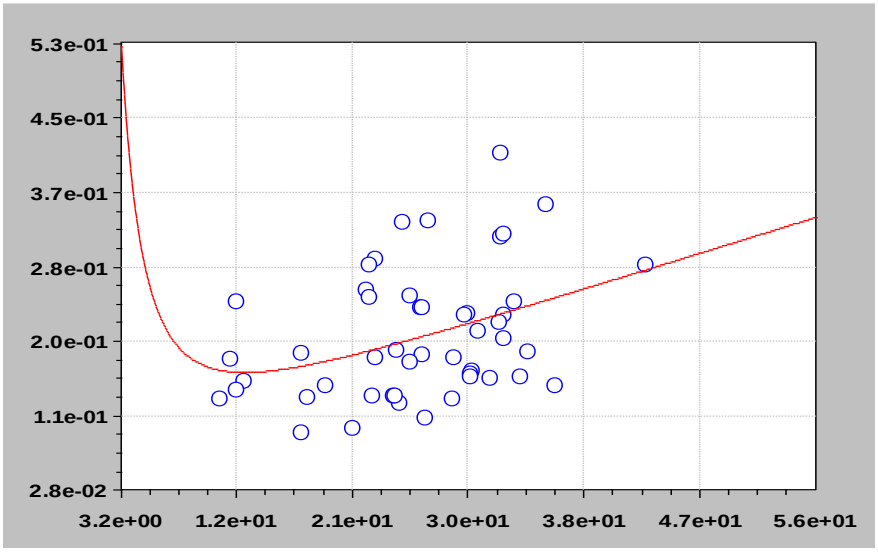


Figure 5.6: Correlation plot between II B Slope and Volatile Matter Content

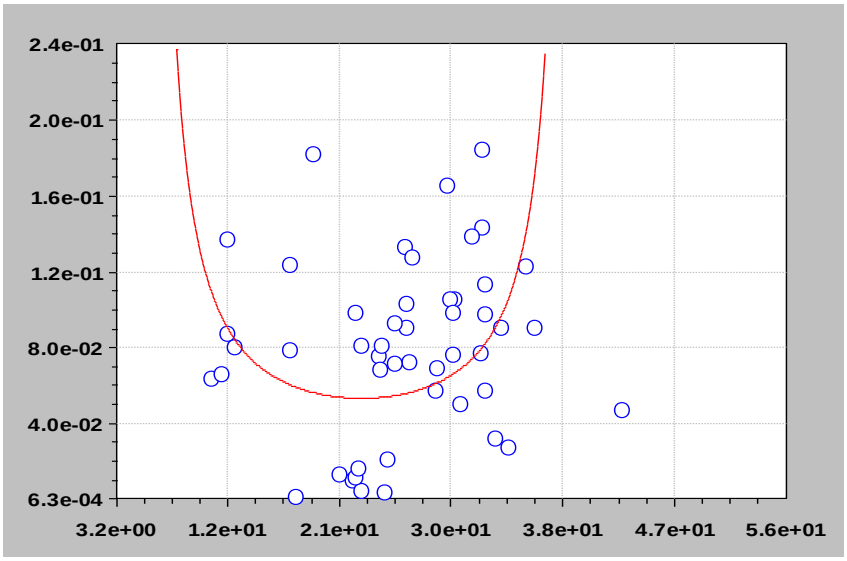


Figure 5.7: Correlation plot between II Slope and Volatile Matter Content

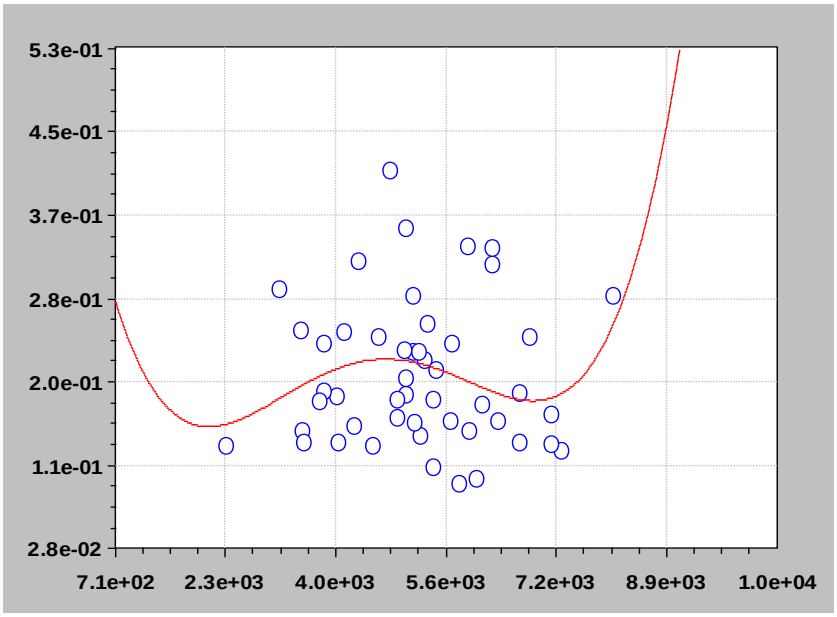


Figure 5.8: Correlation plot between T_c and Ash Content

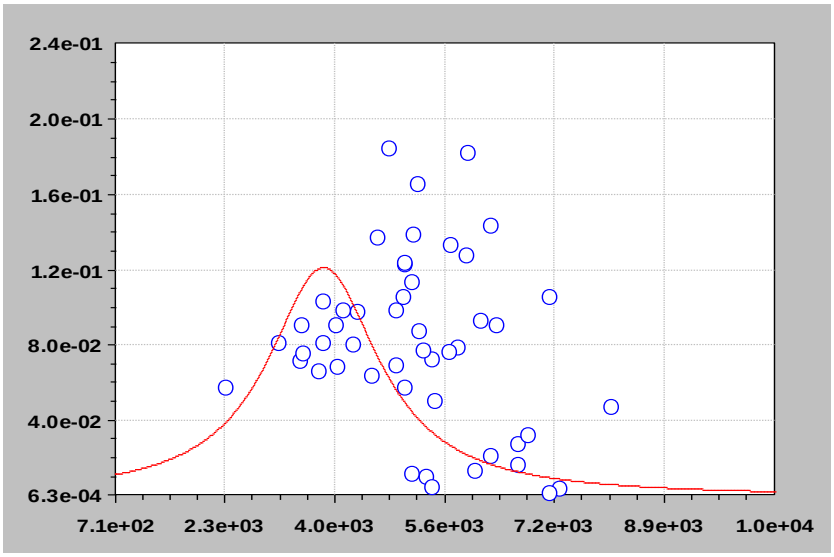


Figure 5.9: Correlation plot between GCV and IIA Slope.

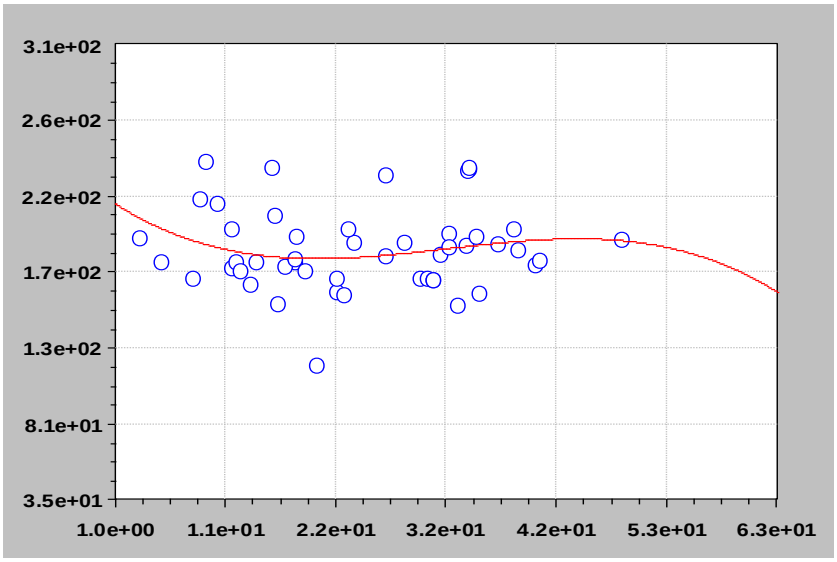


Figure 5.10: Correlation plot between GCV and IIB Slope

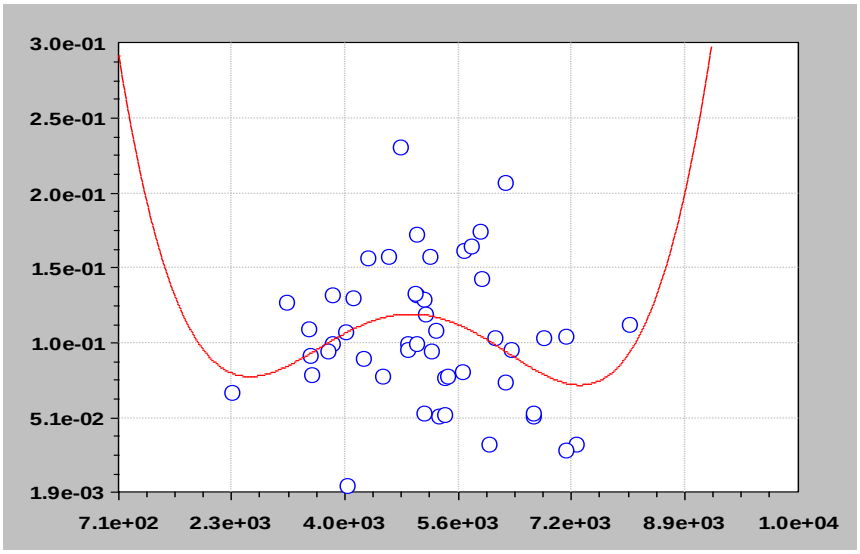


Figure 5.11: Correlation plot between GCV and II Slope

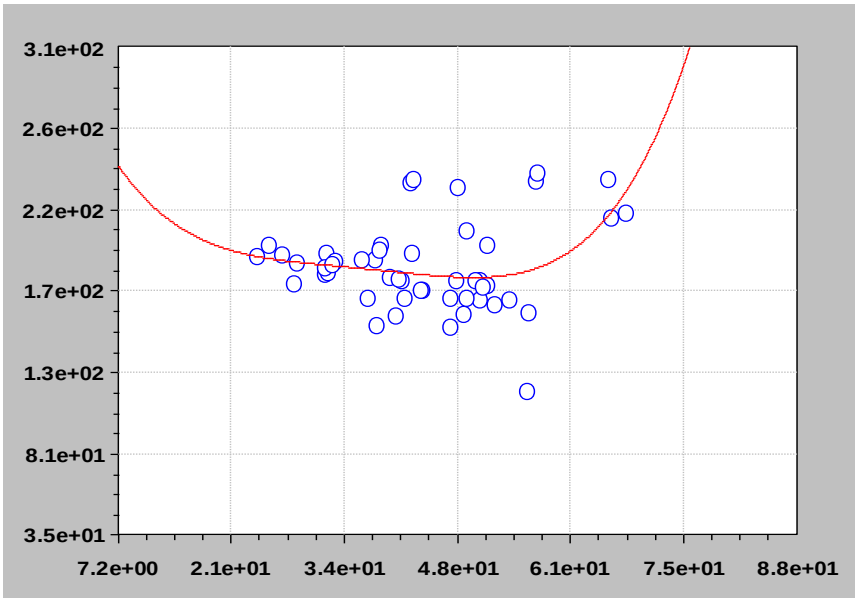


Figure 5.12: Correlation plot between Fixed Carbon and T_c

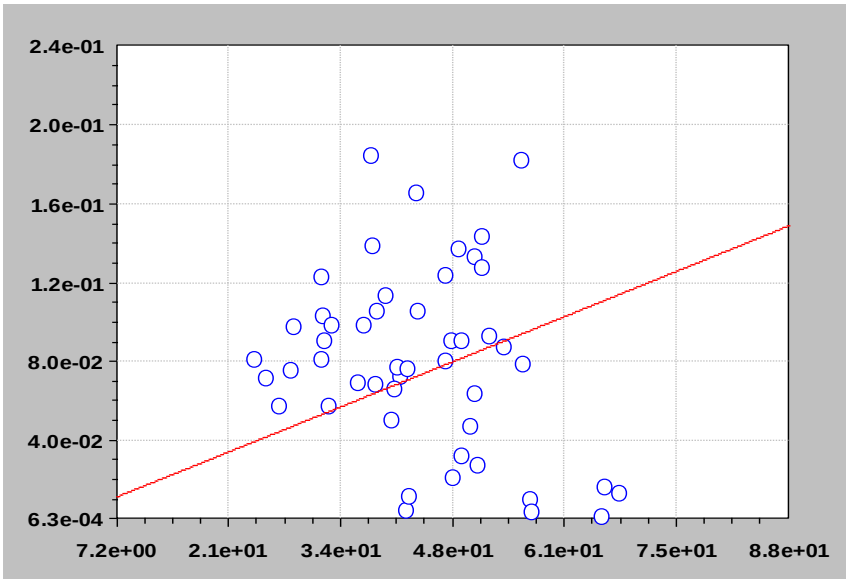


Figure 5.13: Correlation plot between Fixed Carbon and IIA Slope

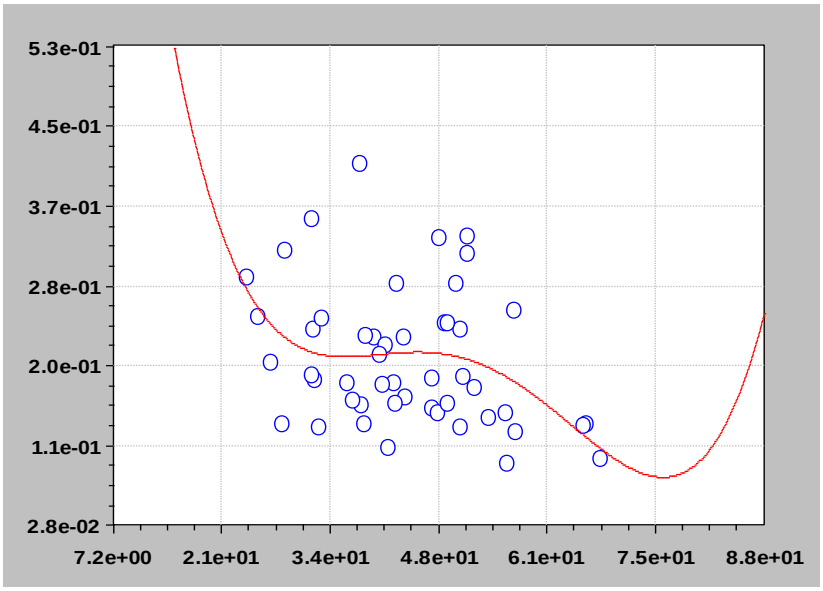


Figure 5.14: Correlation plot between Fixed Carbon and IIB Slope

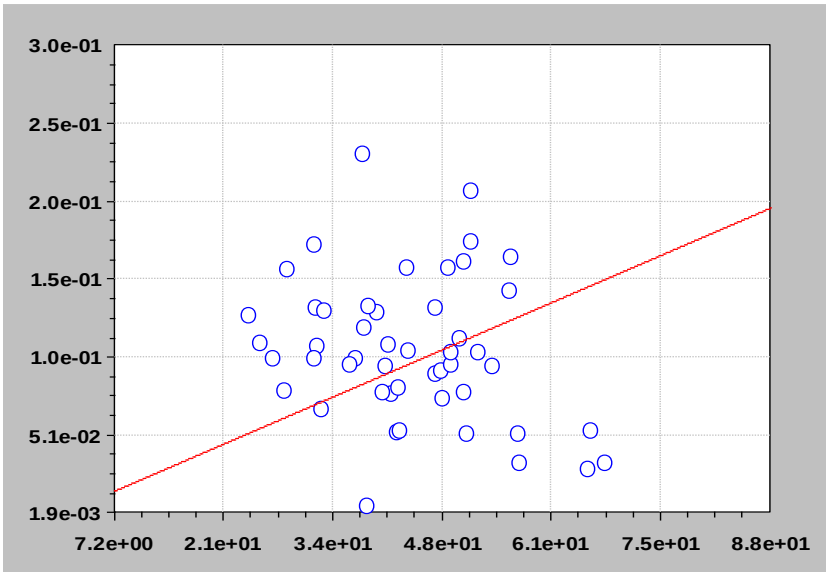


Figure 5.15: Correlation plot between Fixed Carbon and II Slope

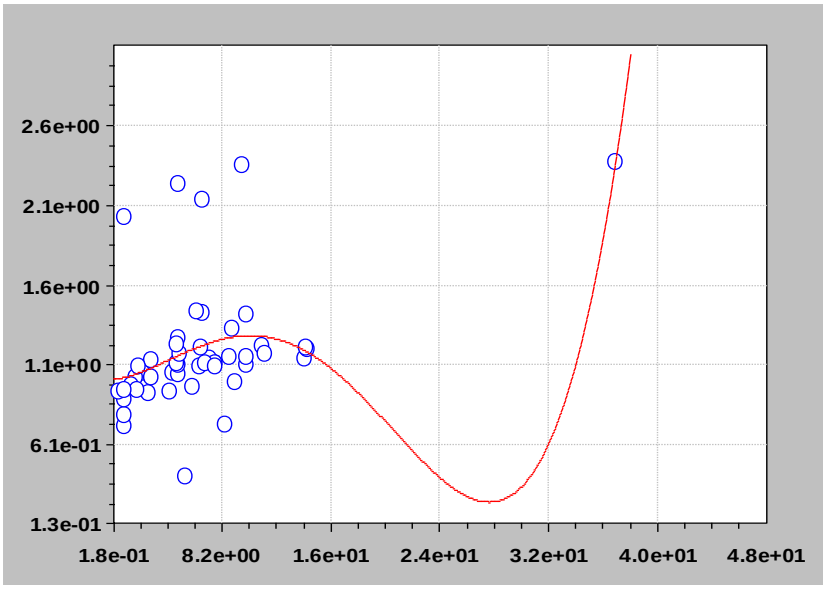


Figure 5.16: Correlation plot between Moisture Content and Aliphaticity

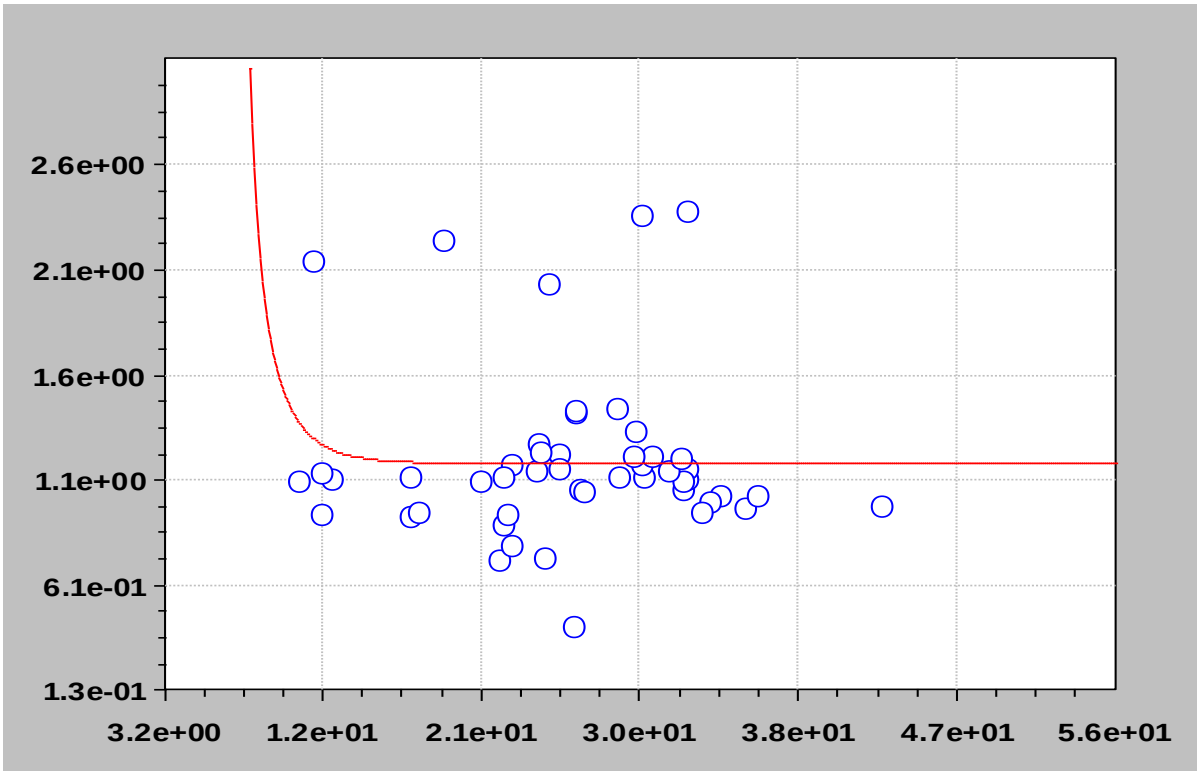


Figure 5.17: Correlation plot between Volatile Matter Content and Aliphaticity

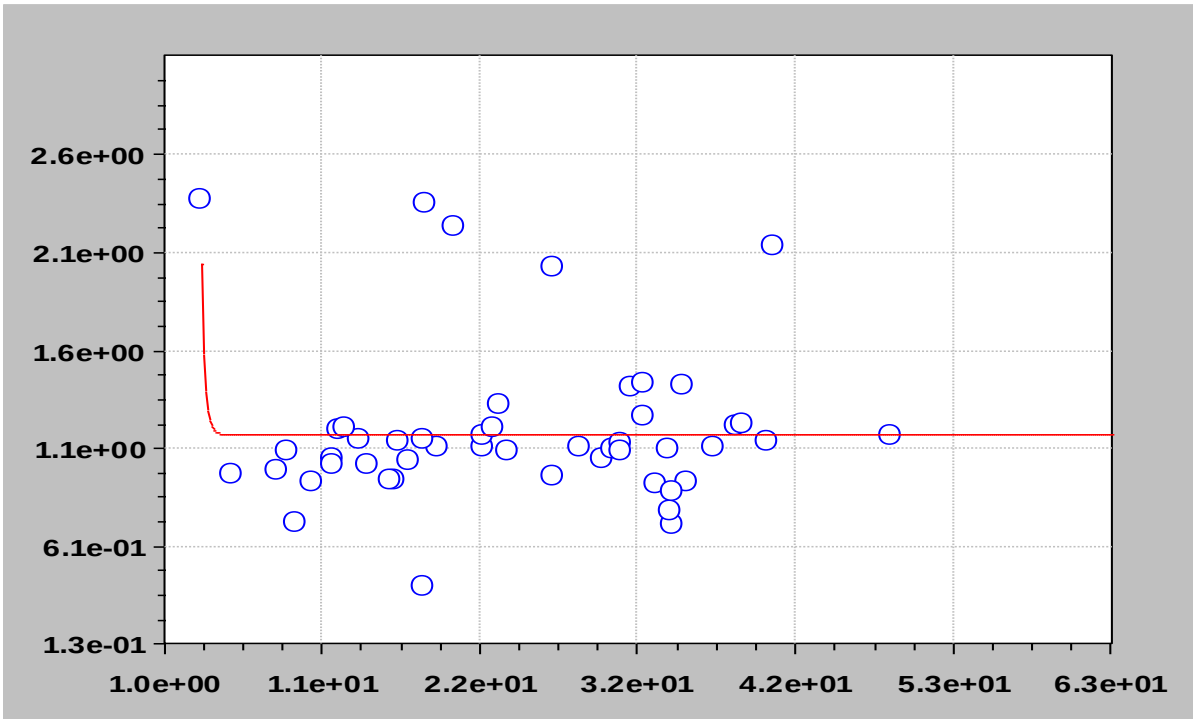


Figure 5.18 Correlation plot between Ash Content and Aliphaticity

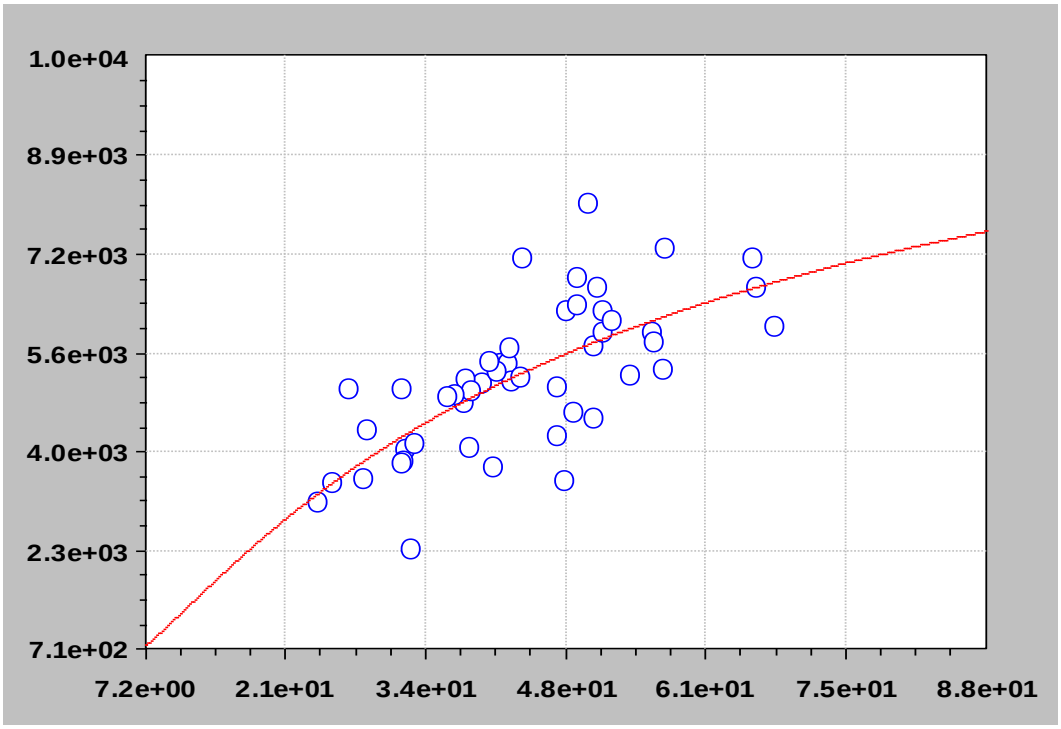


Figure 5.19: Correlation plot between Fixed Carbon and Aliphaticity

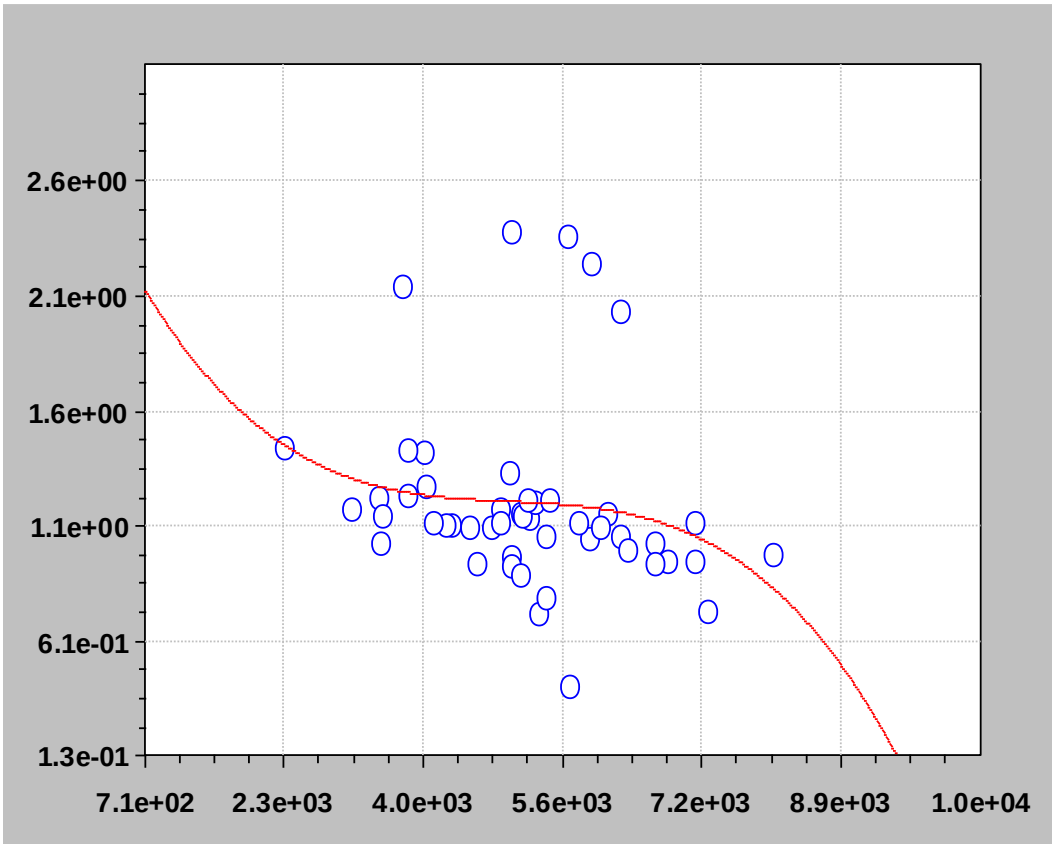


Figure 5.20: Correlation plot between GCV and Aliphaticity

CHAPTER 6

DISCUSSION AND CONCLUSION

6.0 DISCUSSION AND CONCLUSION

6.1 DISCUSSION

In order to assess the spontaneous heating tendency of Indian coals, 49 coal samples belonging to all major coalfields were collected. Their intrinsic properties were determined by proximate analysis ad bomb calorimetry, aliphaticity were determined by FTIR techniques, mineralogy study was done by using XRD technique and spontaneous heating tendency by differential thermal analysis method.

It may be noted from Table 4.2 that the moisture content of coal samples varied from 0.6 (BCCL-2) to 37.30 % (NLC-1). The volatile matter content varied from 10.80 (SECL-7) to 43.03% (NEC-1). The NEC coal has highest volatile matter content followed by NCL, SCCL , MCL and WCL. The MCL coals have high to medium volatile matter content. Similarly the maximum ash content is in CCL-1 coal (48.51%) and lowest ash 3.40% in NEC-1 sample. The NEC sample has the highest Gross calorific value with 8075.08cal/gm, while the lowest calorific value is of MCL-15 with 2366.66 cal/gm. MCL coals have medium to high calorific value while SCCL,SECL,ECL and BCCL has high calorific value.

The transition temperature (T_c) value of the coal samples varied from 115.39^{0}C (SCCL-7) to 238.44^{0}C (ECL-5). Panigrahi and Sahu (2004) have found that the coal seams having onset temperature in the range of 122^{0}C to 140^{0}C are highly susceptible to spontaneous heating. As per the classification suggested by Panigrahi and Sahu (2004) sample SCCL-7 is the highest susceptible to spontaneous heating. This is followed by SCCL-6 and MCL-4 with a T_c of 151.70^0 C and 152.74^{0}C respectively. Similarly the samples collected from ECL has lower susceptible to spontaneous heating with high transition temperature value.

The IIA slope values vary from 0.0021 (BCCL-3) to 0.1884 (SCCL-7), IIB slope varies from 0.0938 (SECL-4) to 0.4113 (MCL-1) and overall slope of stage II varies from 0.0065 (MCL-13) to 0.2298 (MCL-1). It has been observed in the past that higher is the slope values higher is the spontaneous heating susceptibility of coal. SCCL-7 has the lowest onset temperature and the highest slope values among all the samples. So it may be considered to be very highly susceptible.

It may be noted from the correlation study (Table) that moisture and Gross Calorific Value shows good correlation with the onset temperature and slopes obtained from DTA thermogram, and Volatile matter shows good correlation with the indices obtained from DTA thermogram.

The Aliphaticity varied from 0.73 for ECL-1 to 2.37 for SCCL-7. Thus the coal structure of SCCL-7 contains a high number of aliphatic bonds as compared to aromatic bonds. This makes the SCCL-7 sample to be highly susceptible to spontaneous heating.

From the mineralogical study through XRD analysis it has been seen that most of the mineral that are present in the coal sample are silica based mineral i.e quartz and kaolinite followed by muscovite and orthoclase. Sample from NEC, NLC, NCL and MCL showed the presence of sulphur associated minerals.

6.2 CONCLUSION

From the experimental investigation it was found that the coal samples from SCCL were very highly susceptible to spontaneous heating, whereas the samples from BCCL and ECL were found to less susceptible to spontaneous heating. In general the correlation of the intrinsic properties is better with the susceptibility indices obtained from DTA thermogram, particularly with the onset temperature (Tc) and IIA slope of the DTA parameters. Therefore the onset temperature (Tc) and the IIA slope obtained from DTA thermogram can be adopted as a better measure of spontaneous heating tendency of Indian coals. The correlation of intrinsic properties with the susceptibility indices showed that the properties like moisture, volatile matter and fixed carbon have good correlation with the onset temperature (Tc) and IIA slope. This is consistent with earlier works by other researchers.

In India, crossing point temperature (CPT) is taken as the measure of spontaneous heating susceptibility of coals in India. However, it has been observed in the past that it does not predict the susceptibility for all types of coals, particularly for high moisture coals. Moreover, it takes approximately 3 hours to determine the CPT which is time consuming. In the event of a power failure, the whole experiment has to be repeated. Reproducibility of experimental results of Crossing Point Temperature also posed a major problem in the dissertation. Some of these demerits can be overcome by carrying out the differential thermal analysis, where it takes about 2 hours for the completion of an experiment. The

results are highly reproducible and it is very easy to determine the transition temperature and slope values using the software available with the equipment.

Aliphaticity also showed a good correlation with both the above parameter used and therefore it is concluded that it can be used for assessment of spontaneous heating liability of coal. The use of aliphaticity over other properties for prediction of spontaneous heating liabilities is also supported by the fact that the process of measuring aliphaticity by FTIR analysis is very accurate and results are reproducible.

CHAPTER 7

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7. REFERENCES

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APPENDIX-1

DTA Thermograms for Coal Samples

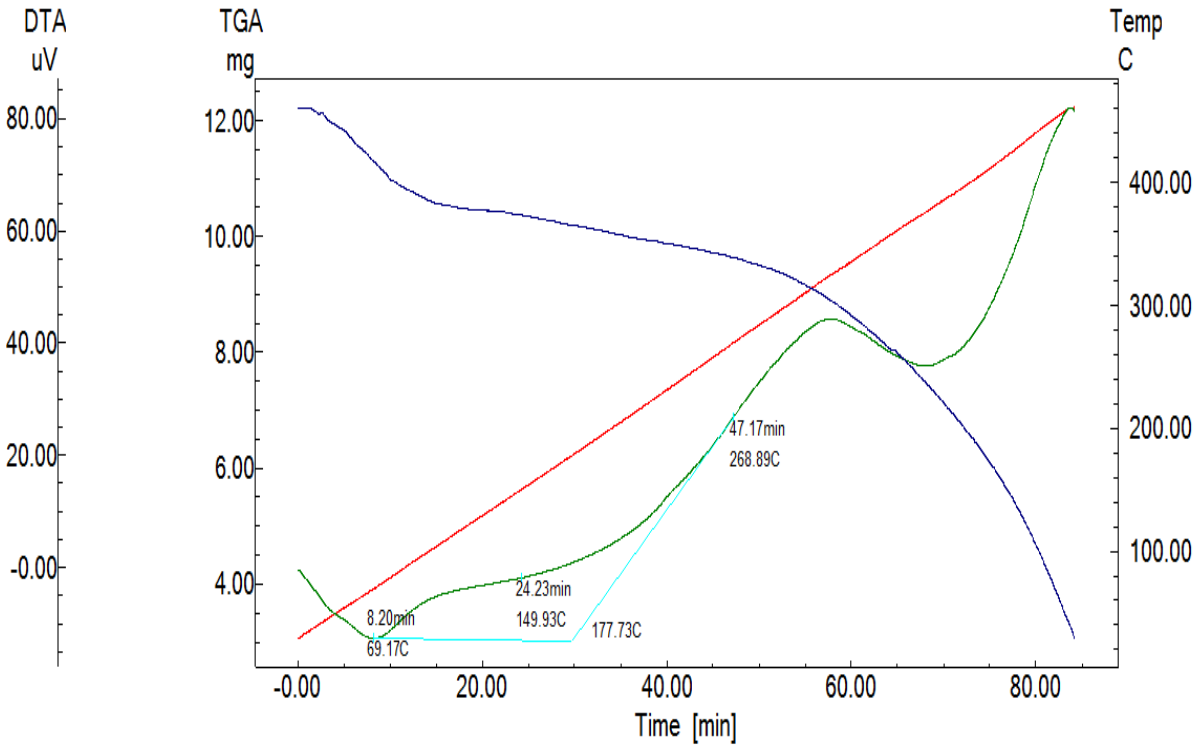


Figure A1-1: DTA Thermogram for SCCL-1

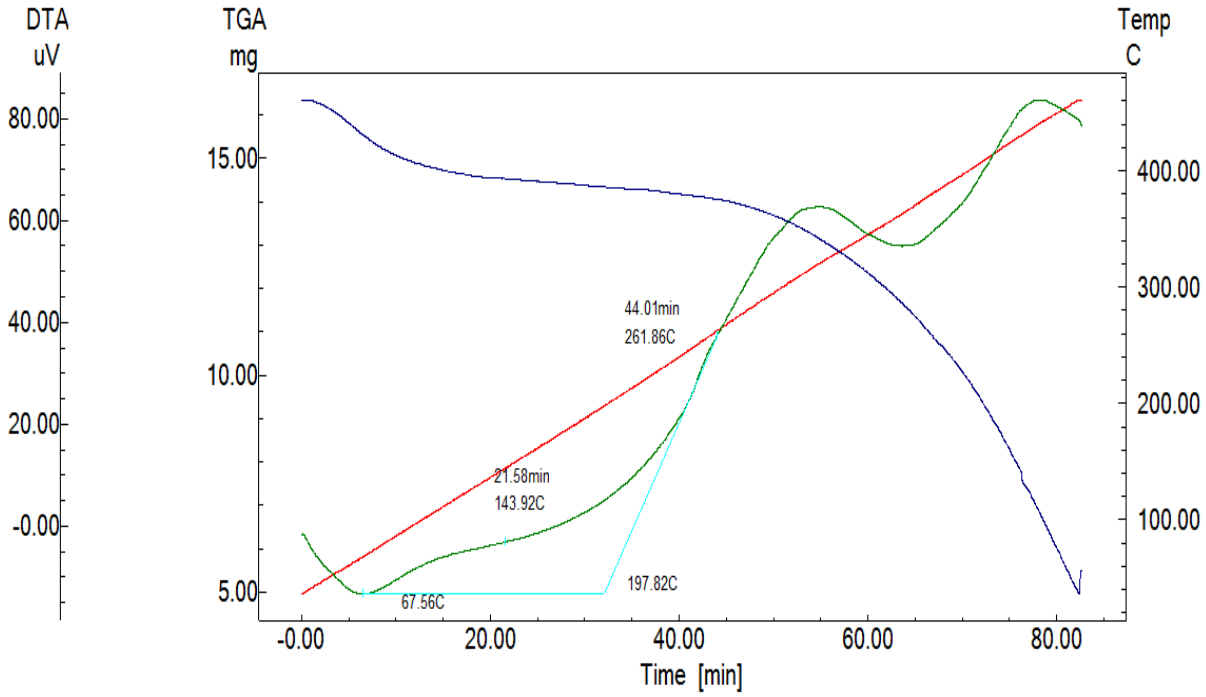


Figure A1-2:DTA Thermogram for SCCL-2

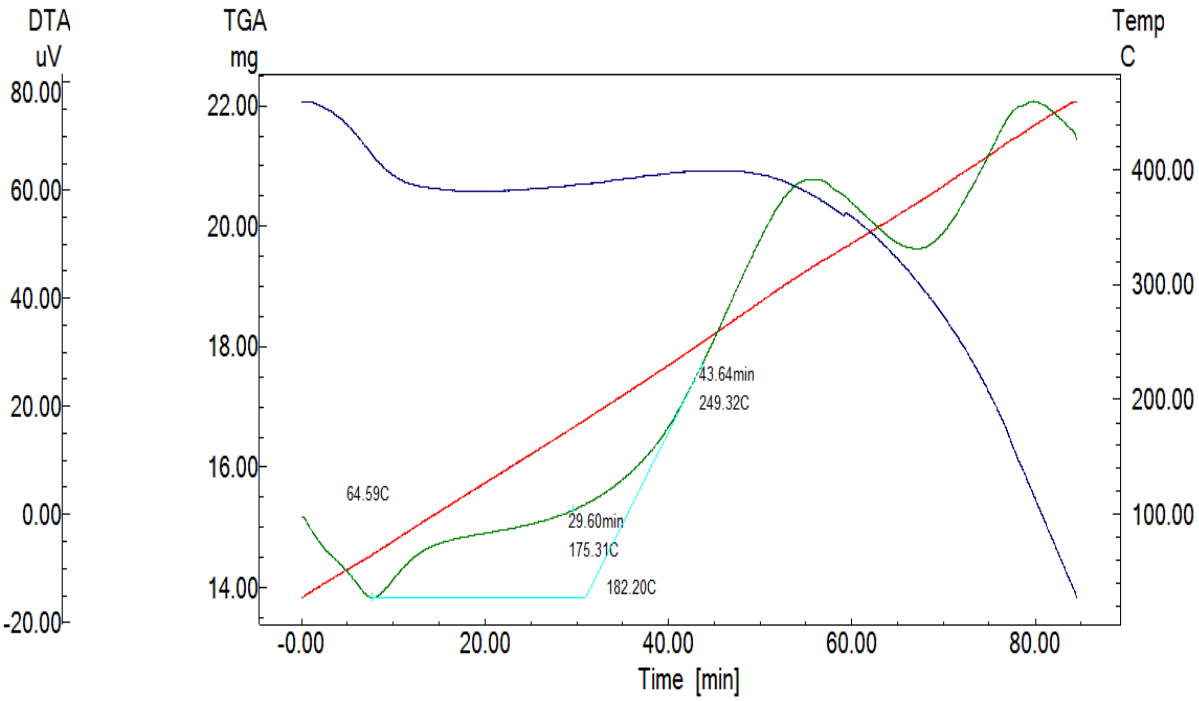


Figure A1-3: DTA Thermogram for SCCL-3

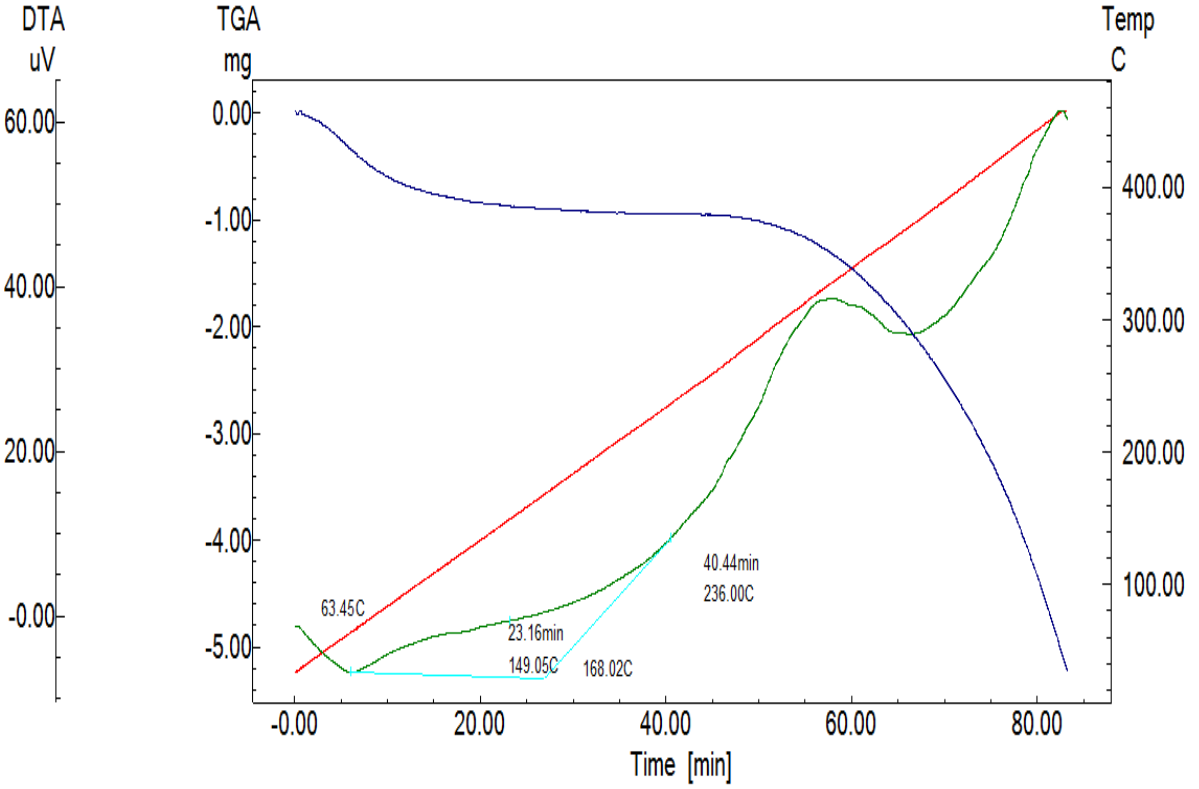


Figure A1-4: DTA Thermogram for SCCL-4

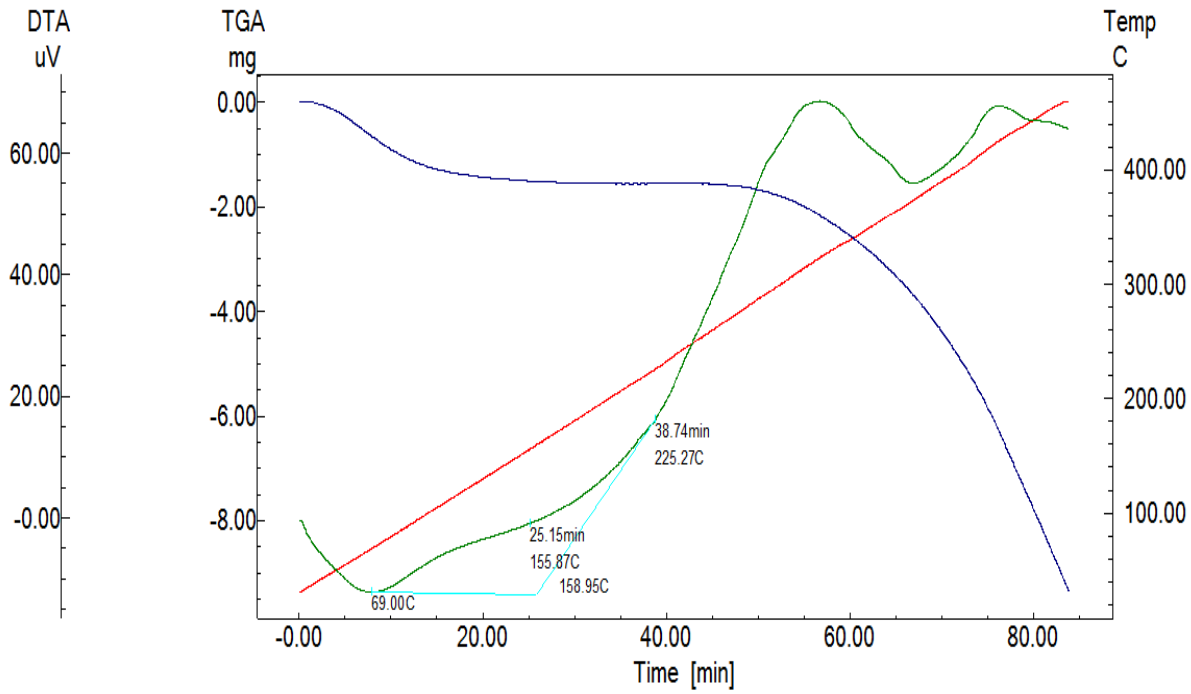


Figure A1-5: DTA Thermogram for SCCL-5

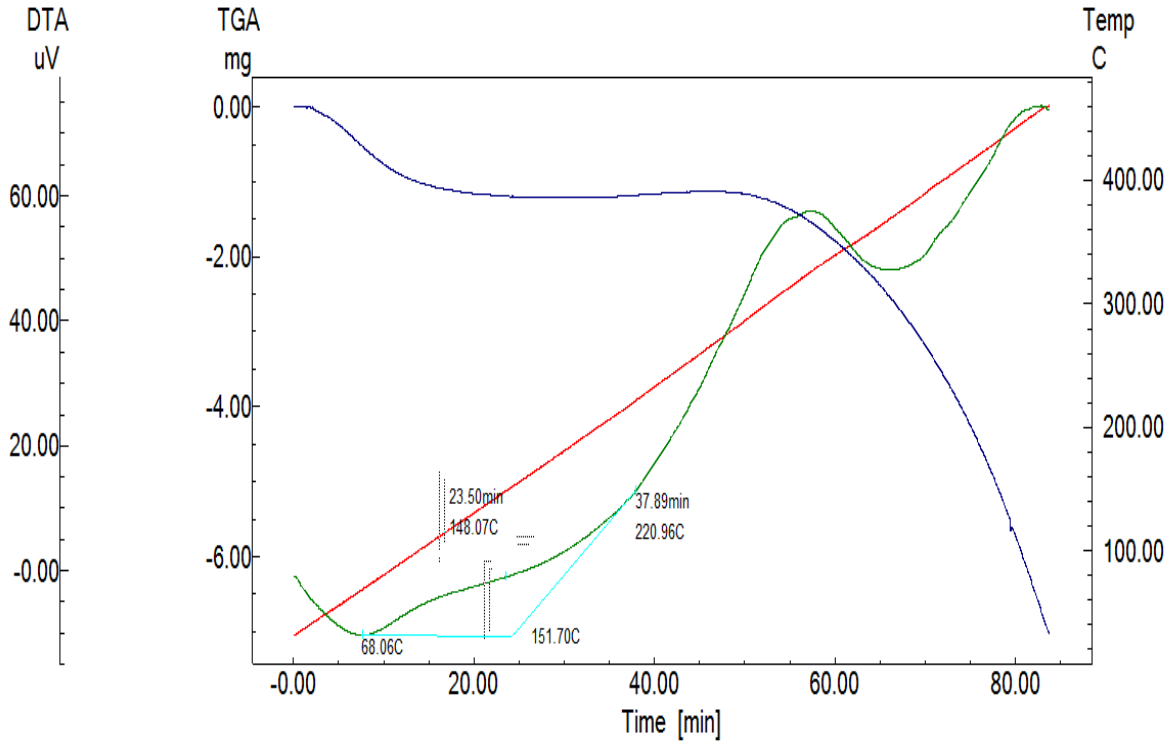


Figure A1-6: DTA Thermogram for SCCL-6

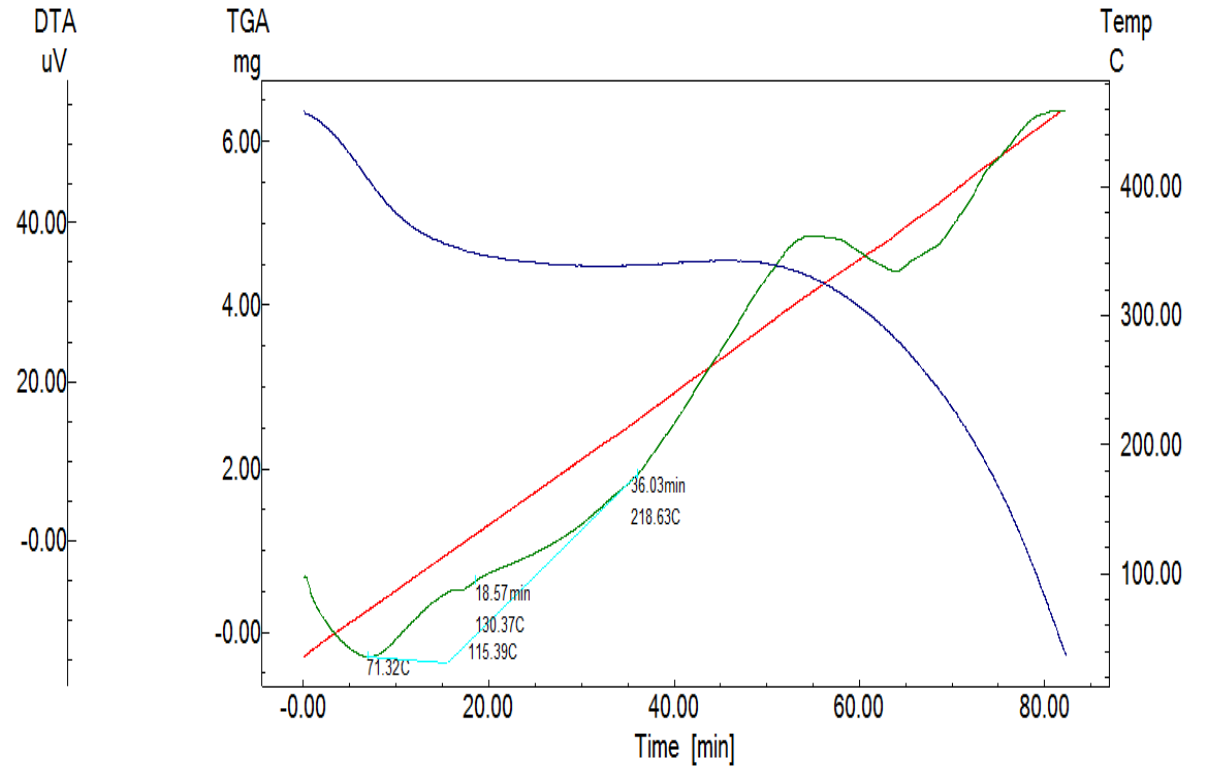


Figure A1-7: DTA Thermogram for SCCL-7

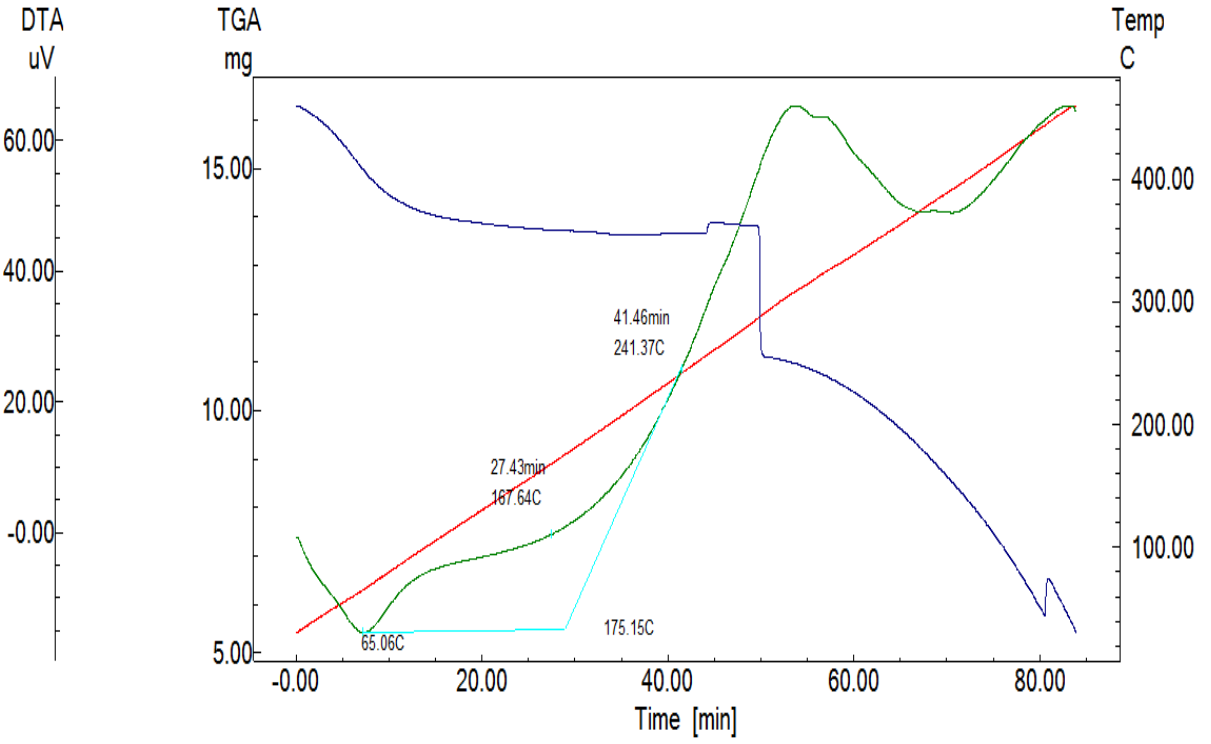


Figure A1-8: DTA Thermogram for SECL-1

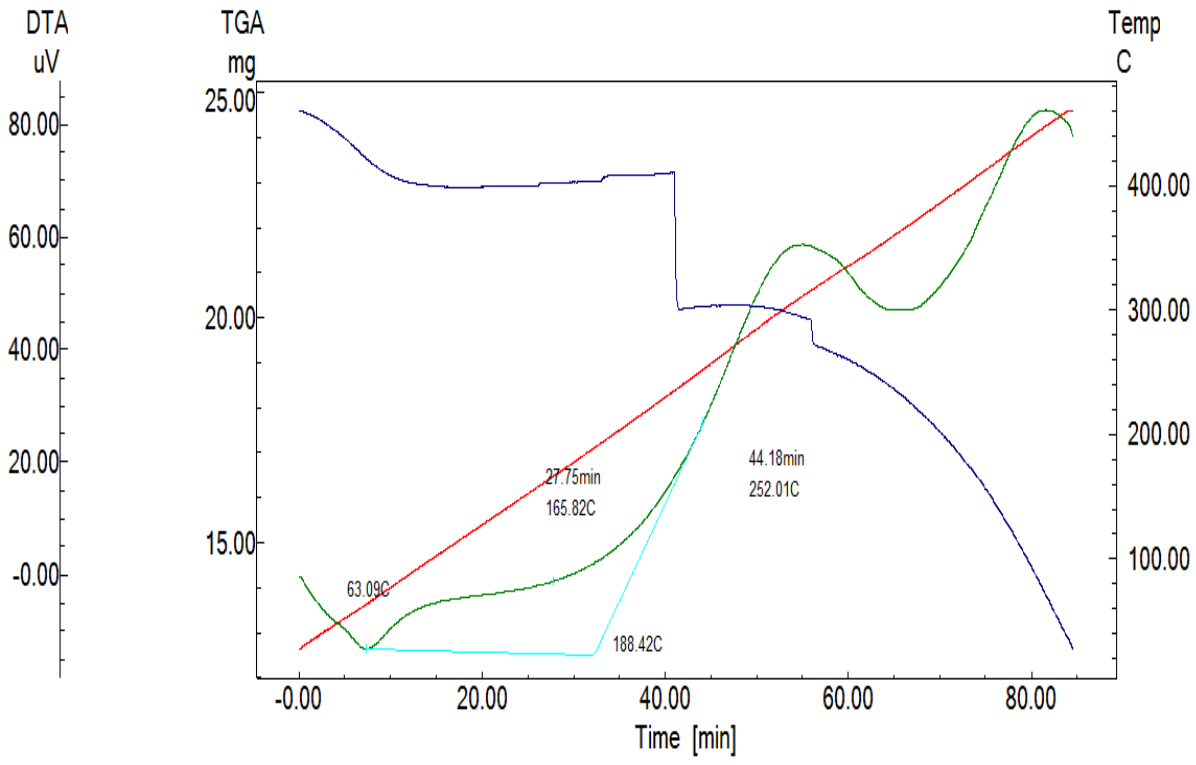


Figure A1-9: DTA Thermogram for SECL-2

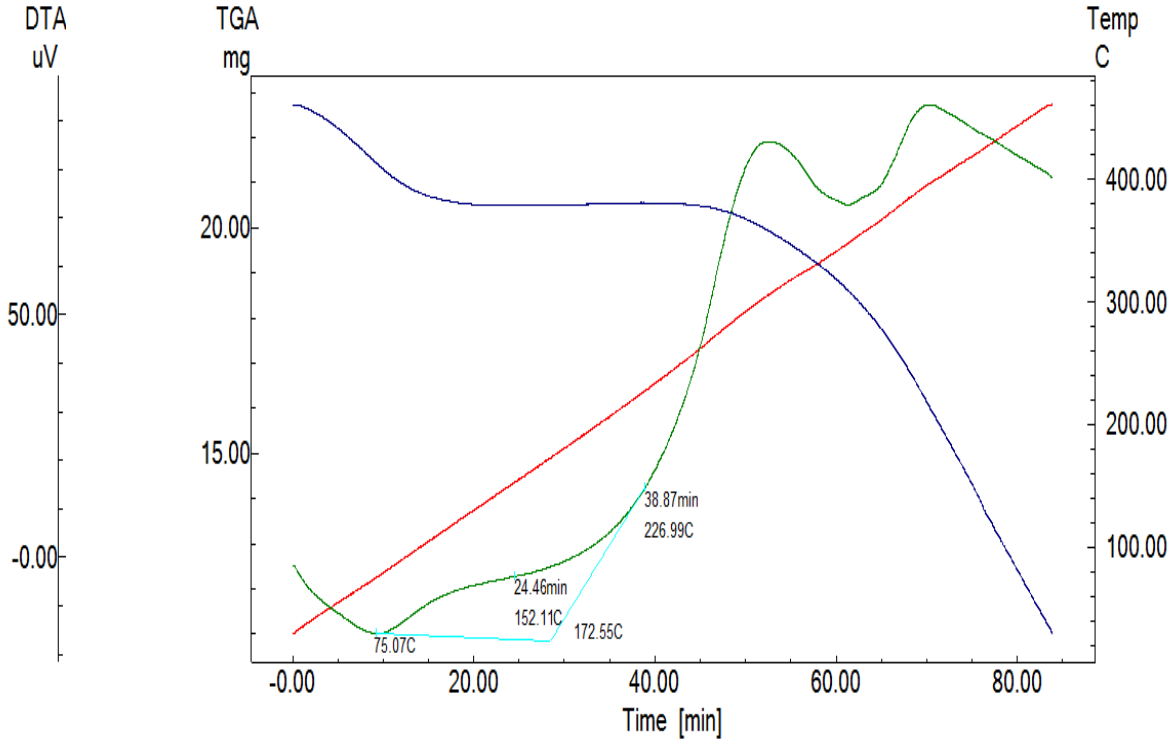


Figure A1-10:DTA Thermogram for SECL-3

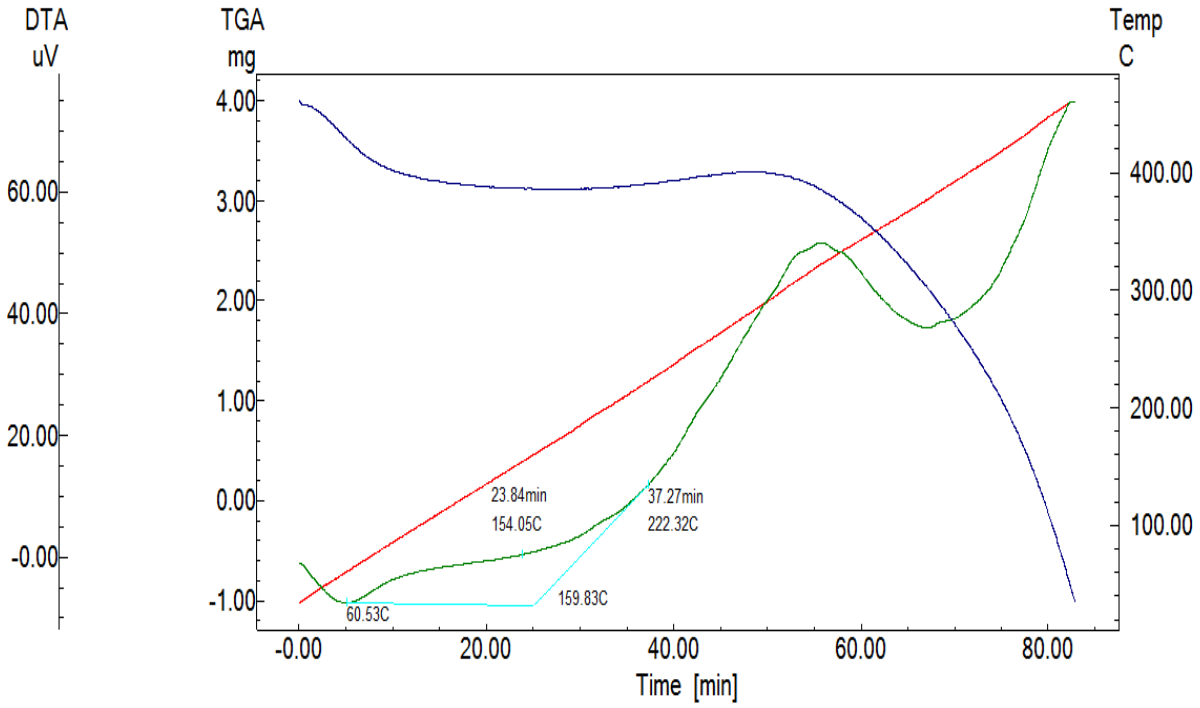
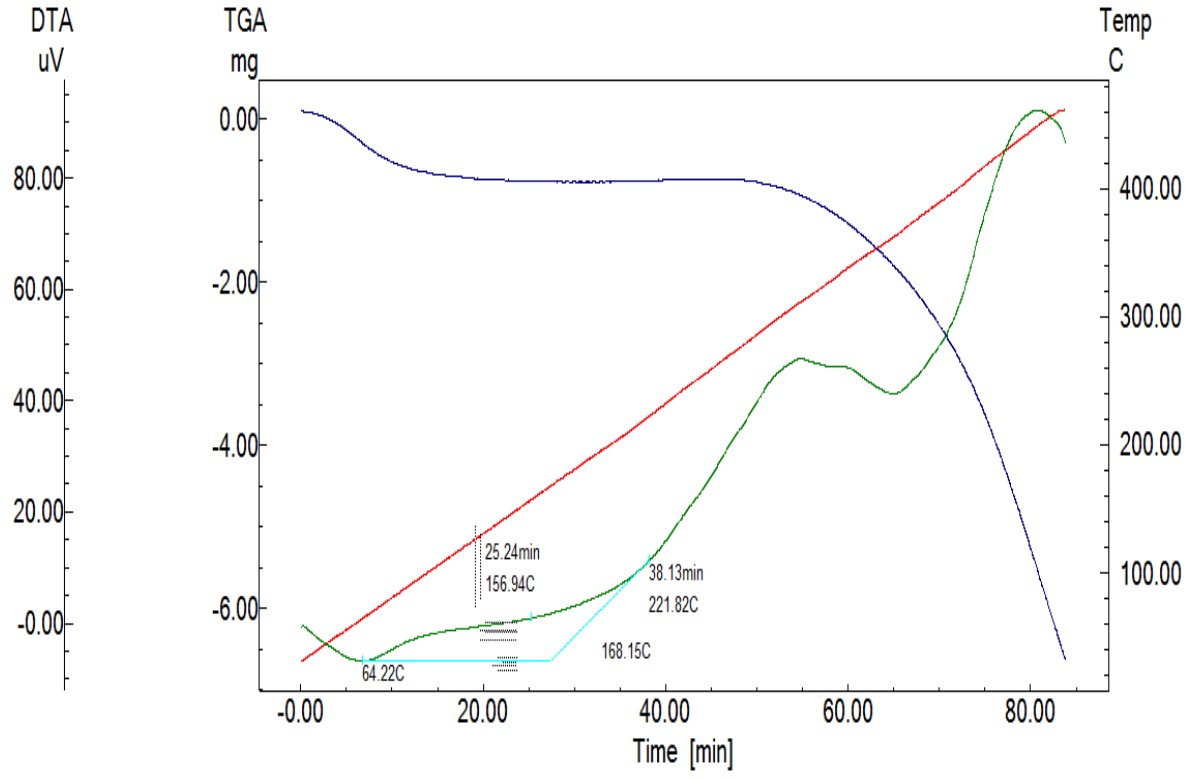


Figure A1-11: DTA Thermogram for SECL-4



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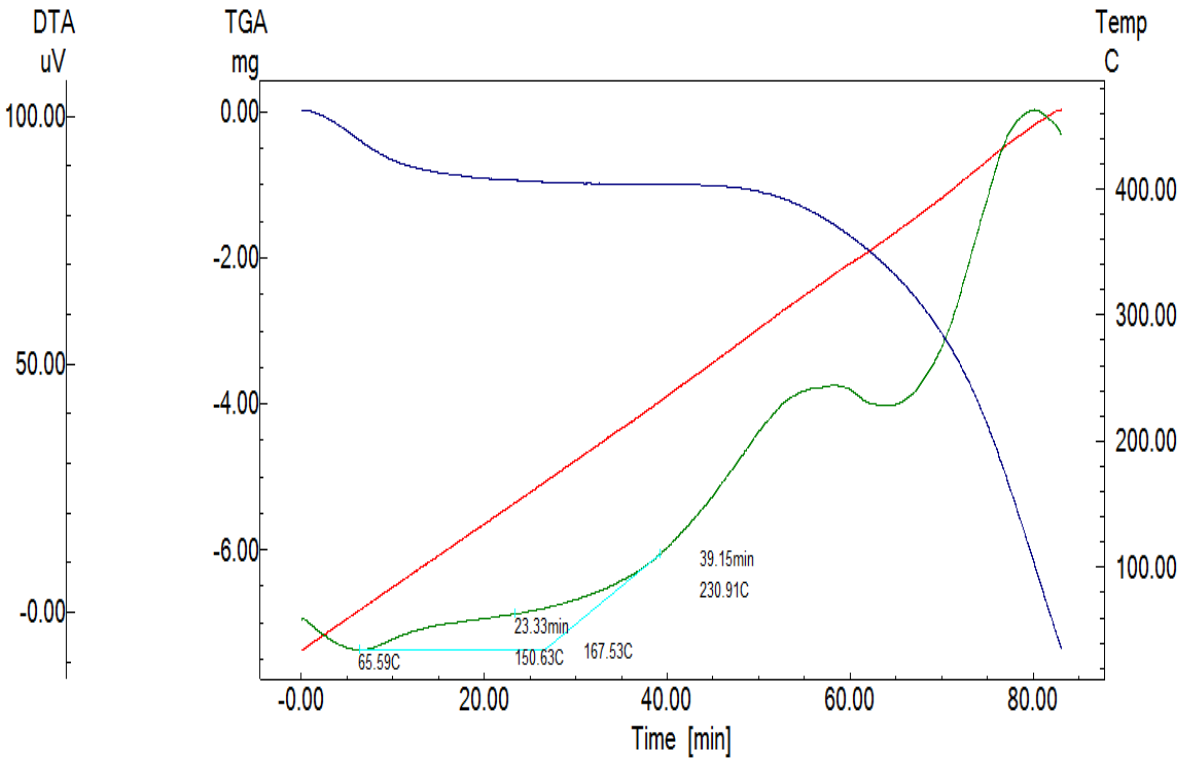

Figure A1-13: DTA Thermogram For SECL-6

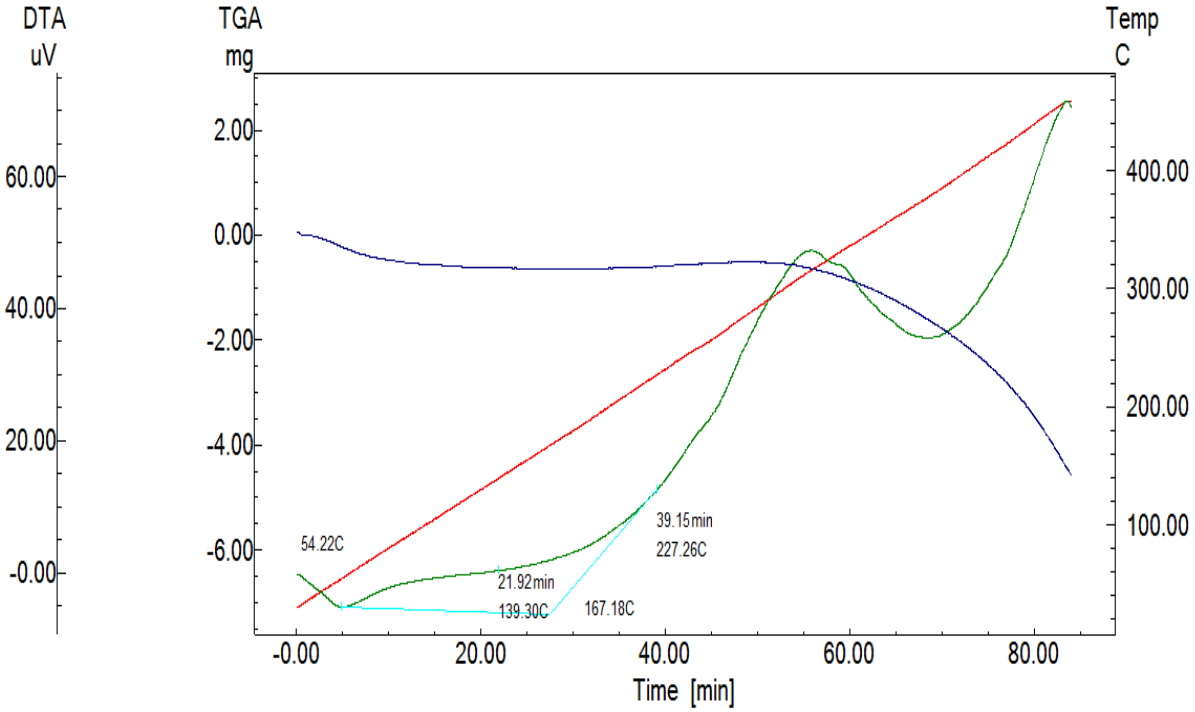

Figure A1-14: DTA Thermogram For SECL-7

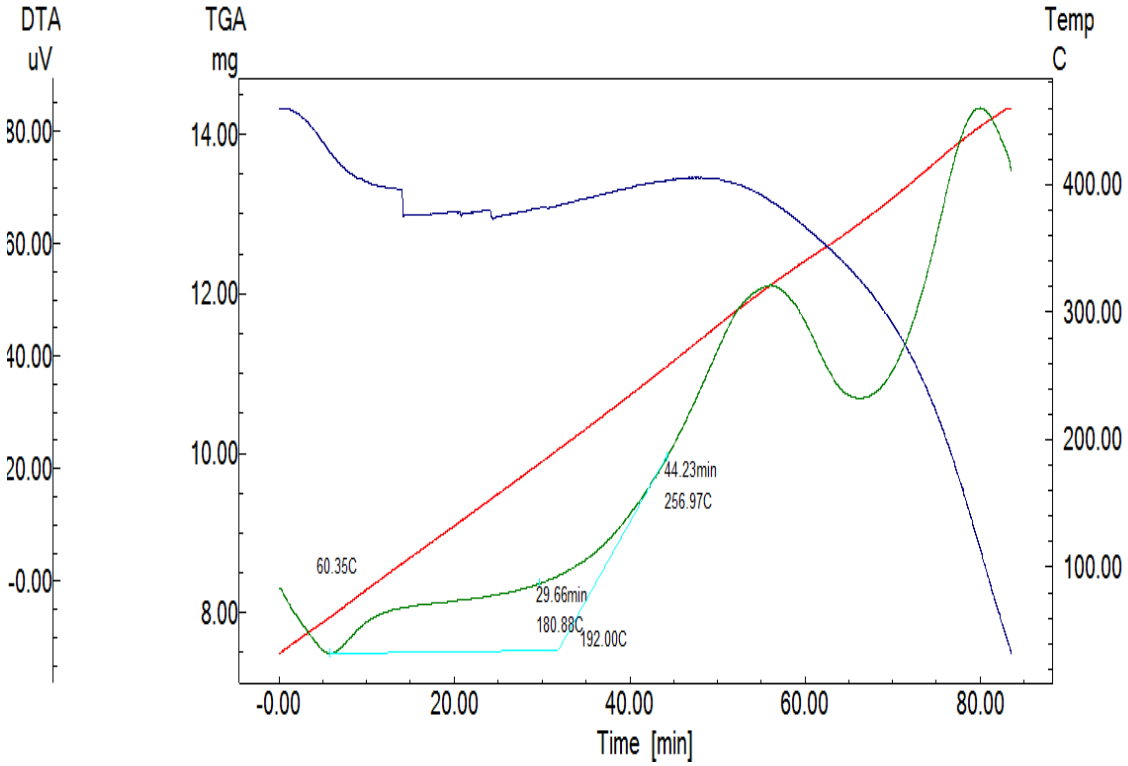

Figure A1-15: DTA Thermogram For CCL-1

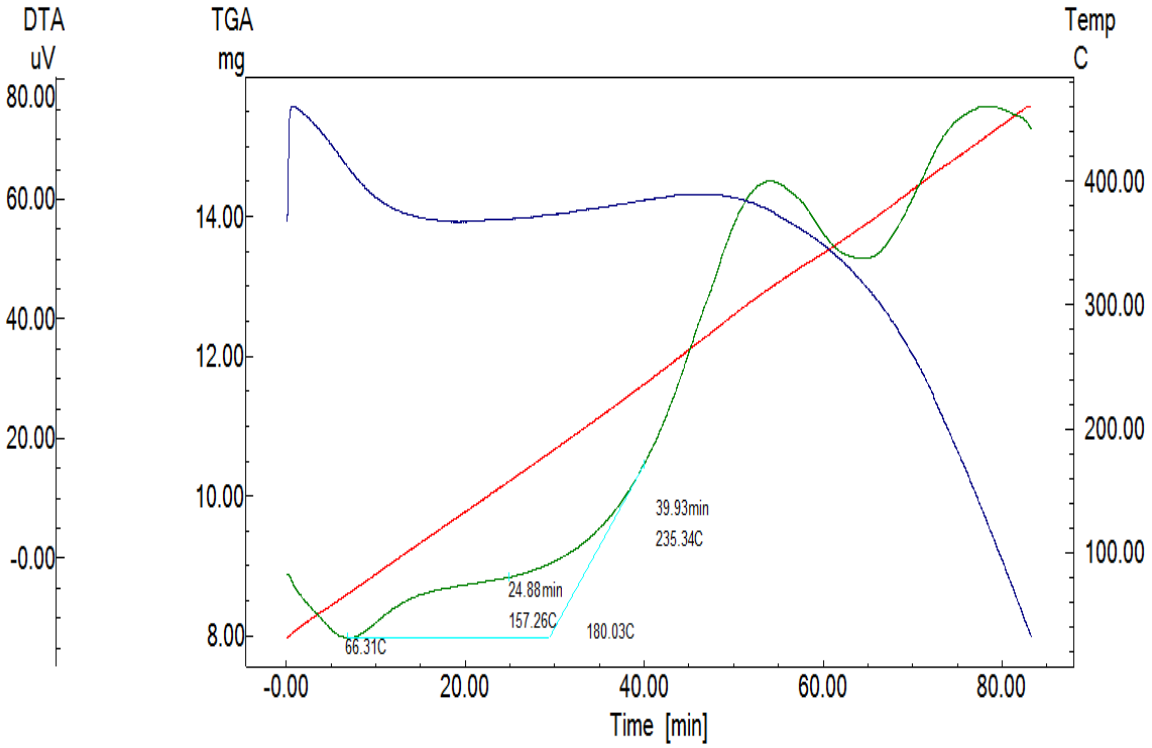

Figure A1-16: DTA Thermogram For CCL-2

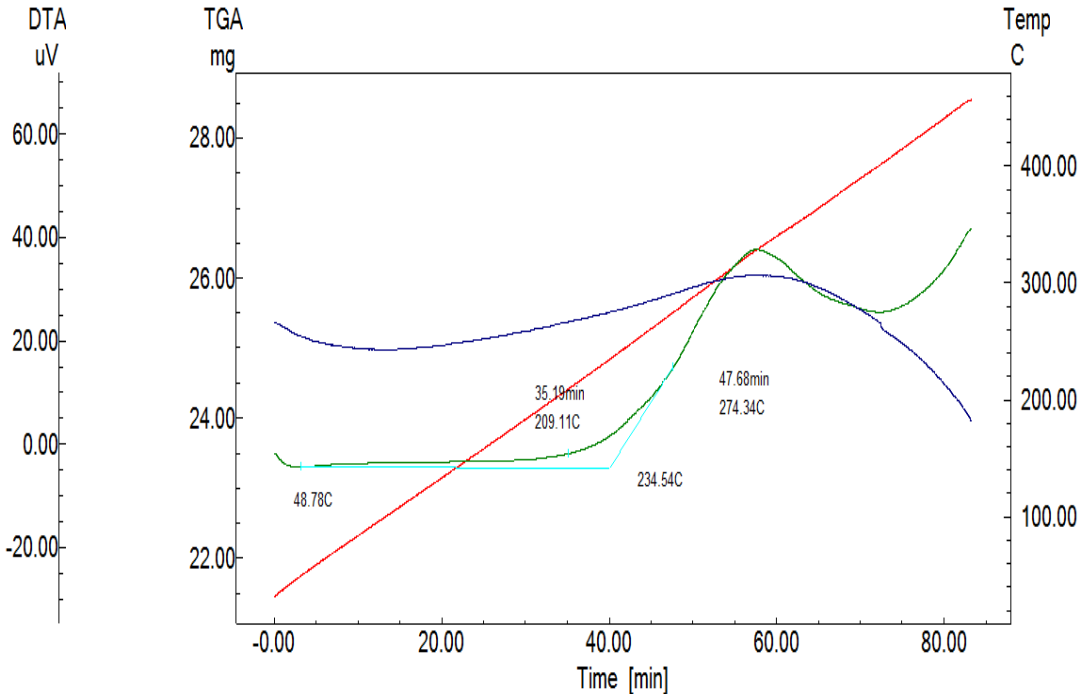

Figure A1-17: DTA Thermogram For ECL-1

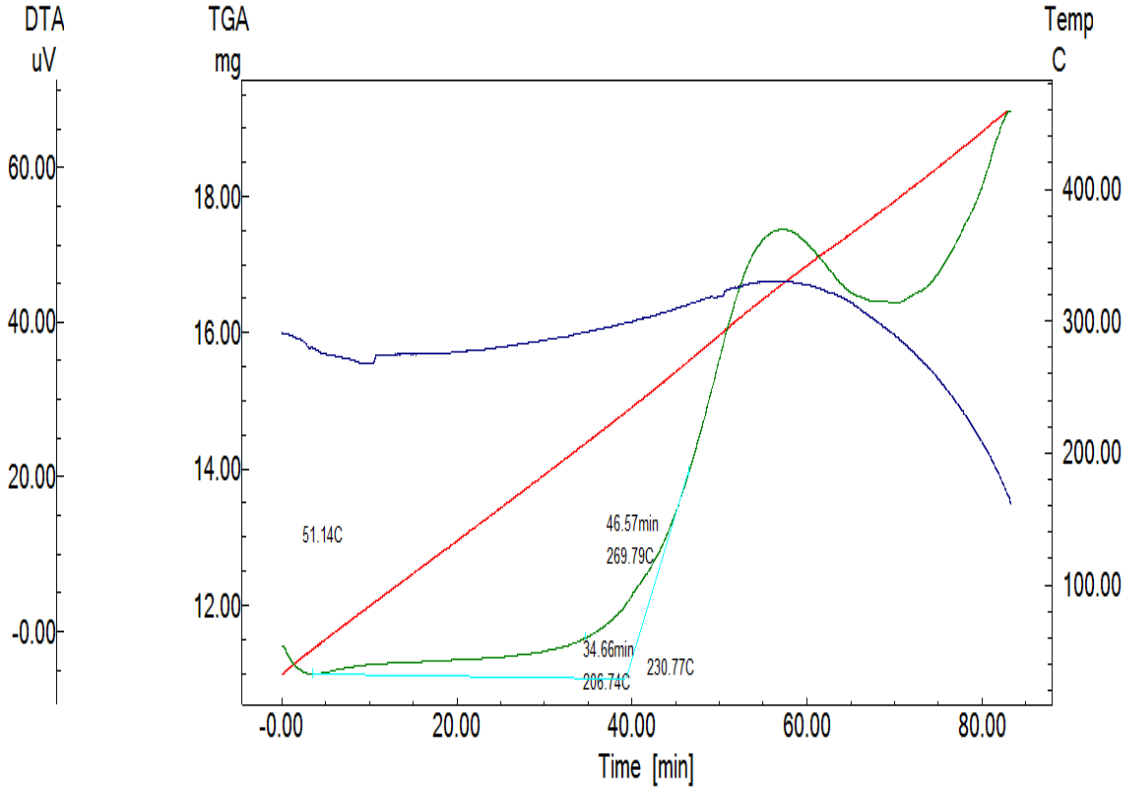

Figure A1-18: DTA Thermogram For ECL-2

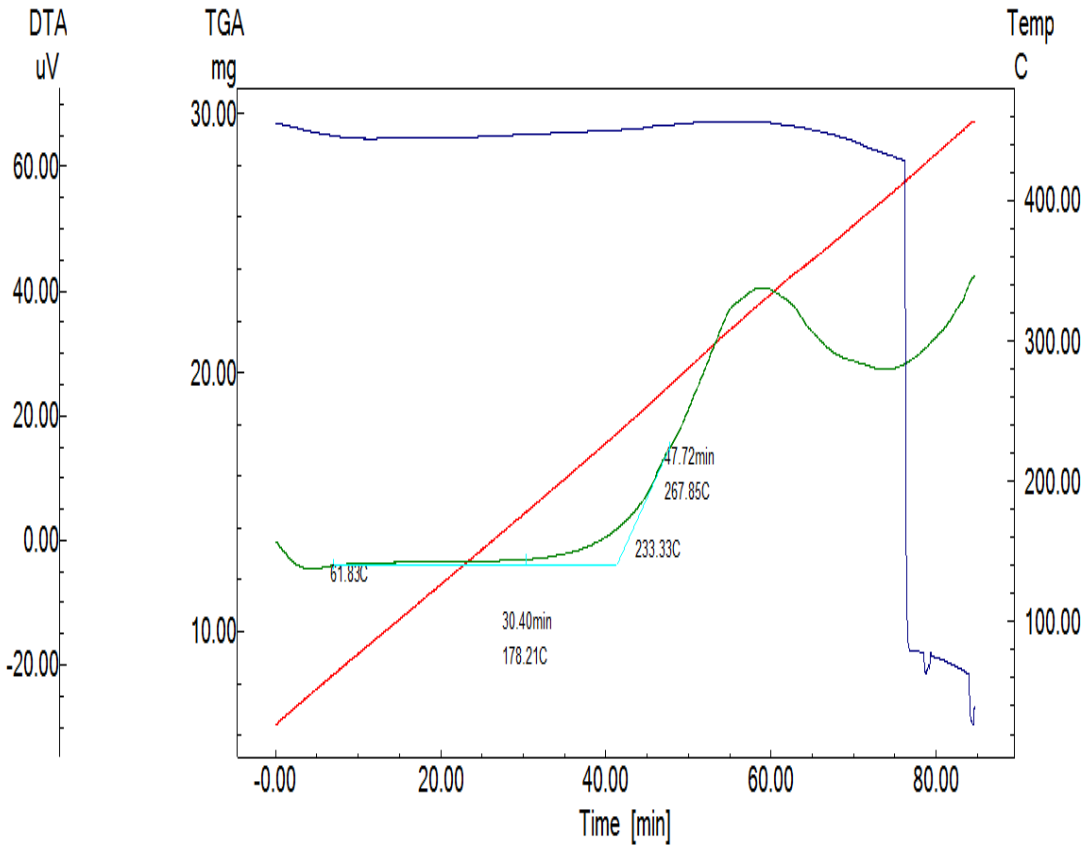

Figure A1-19: DTA Thermogram For ECL-3

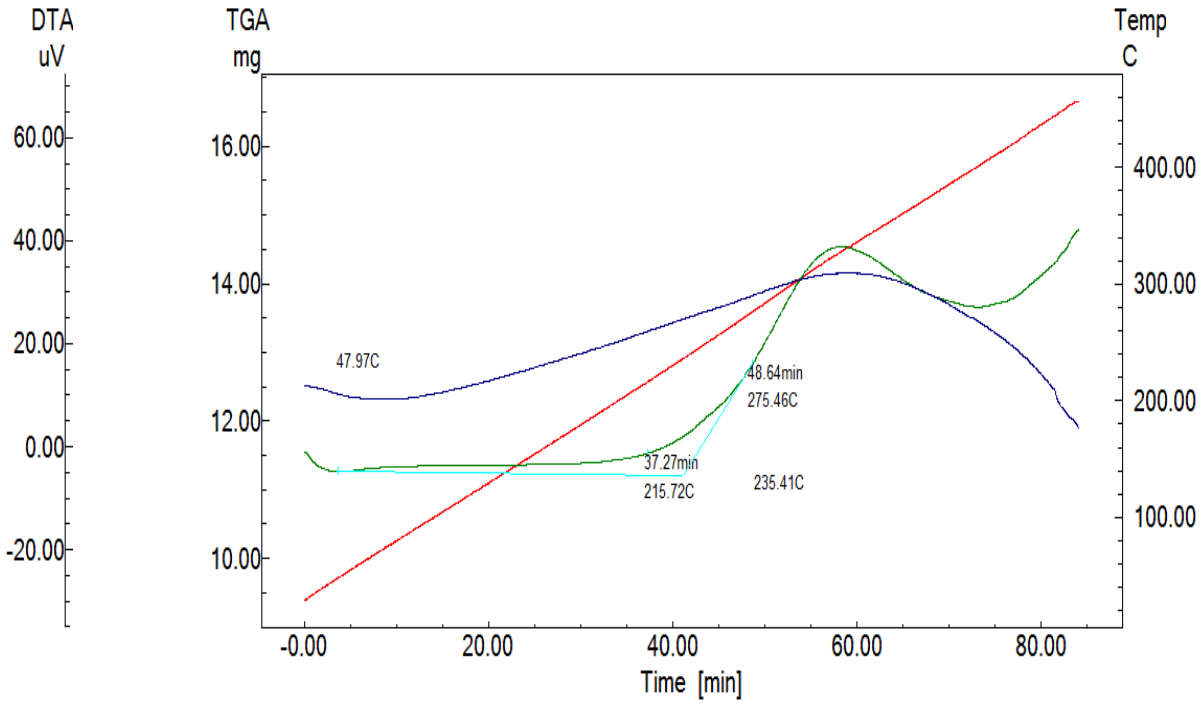

Figure A1-20: DTA Thermogram For ECL-4

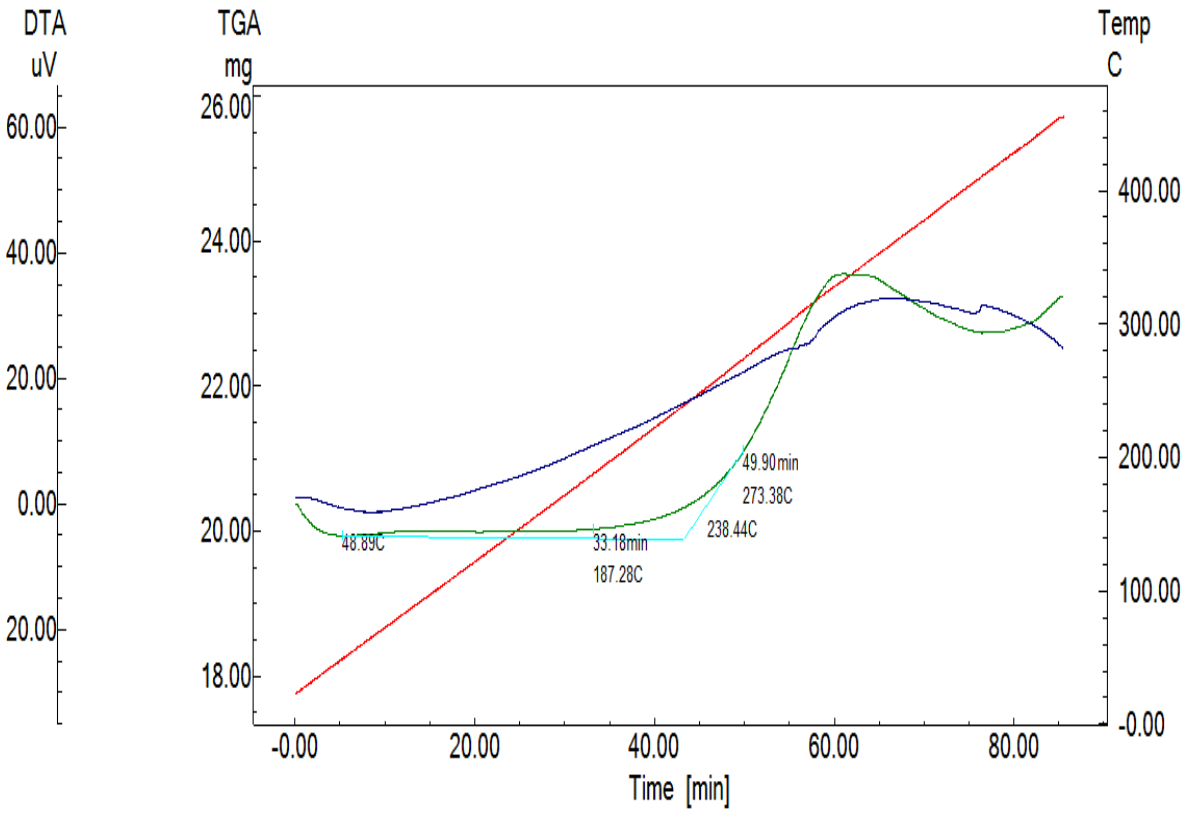

Figure A1-21: DTA Thermogram For ECL-5

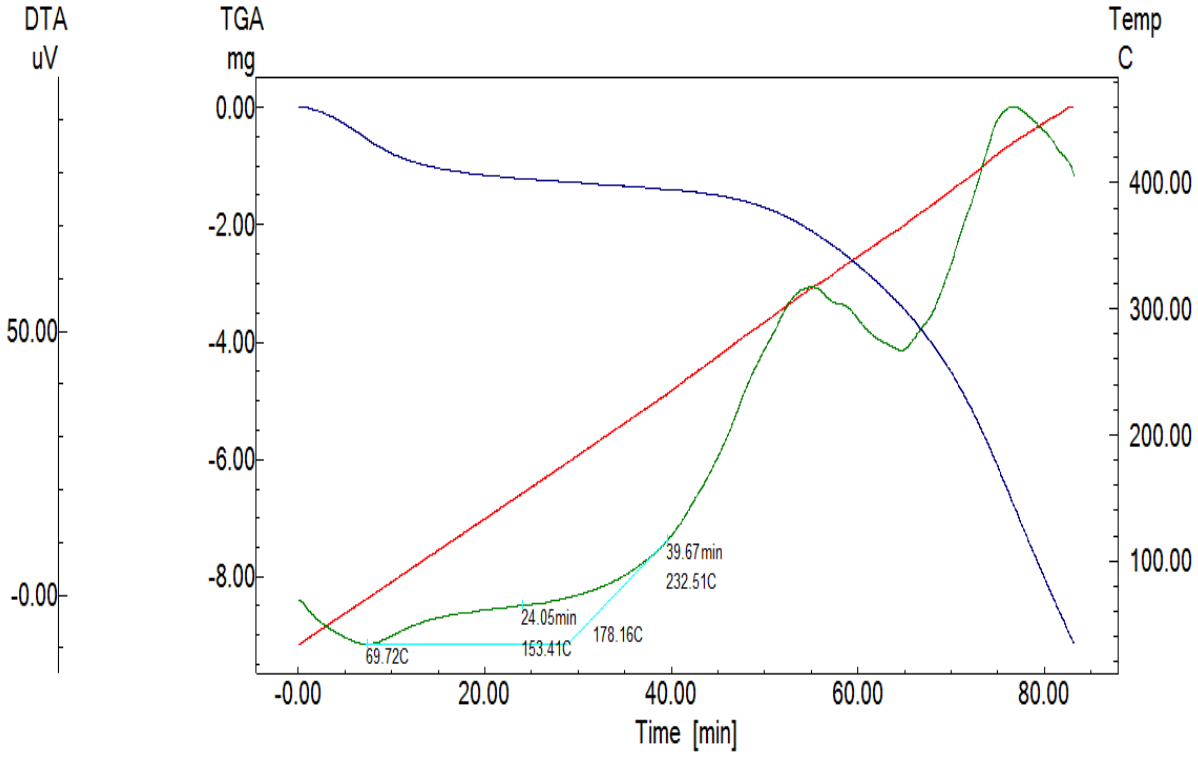

Figure A1-22: DTA Thermogram For ECL-6

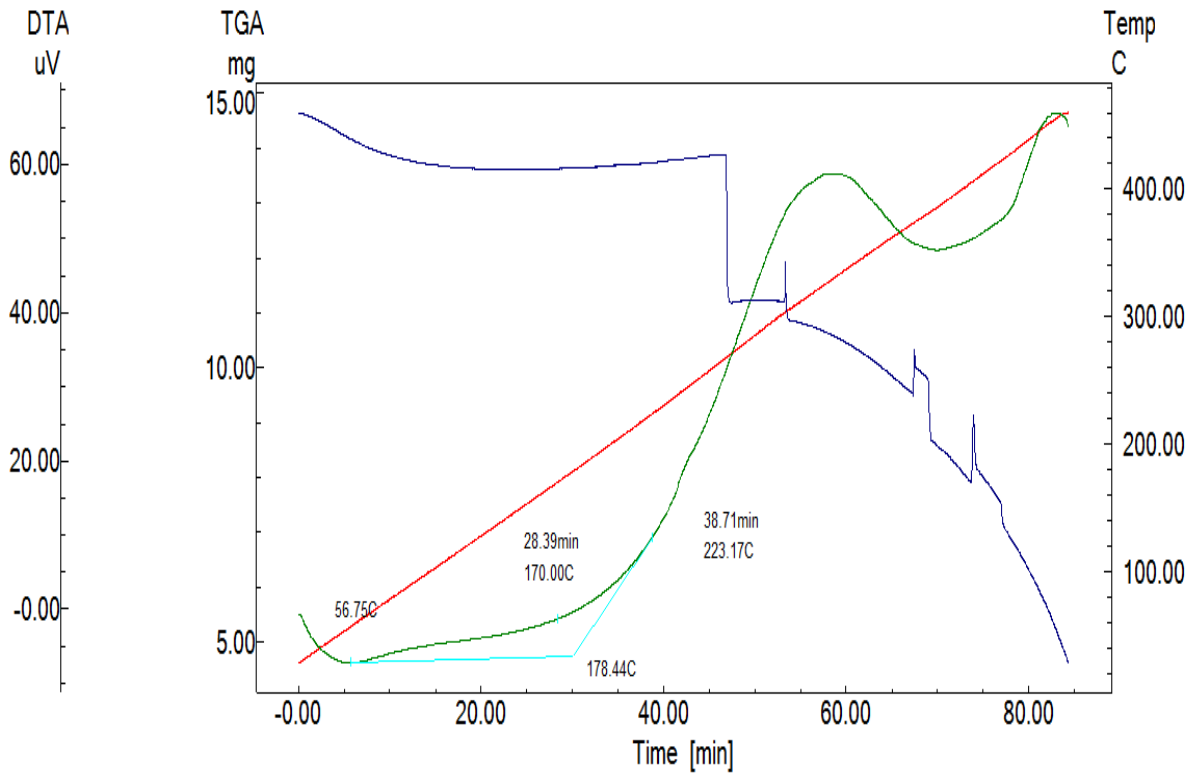

Figure A1-23: DTA Thermogram For NEC-1

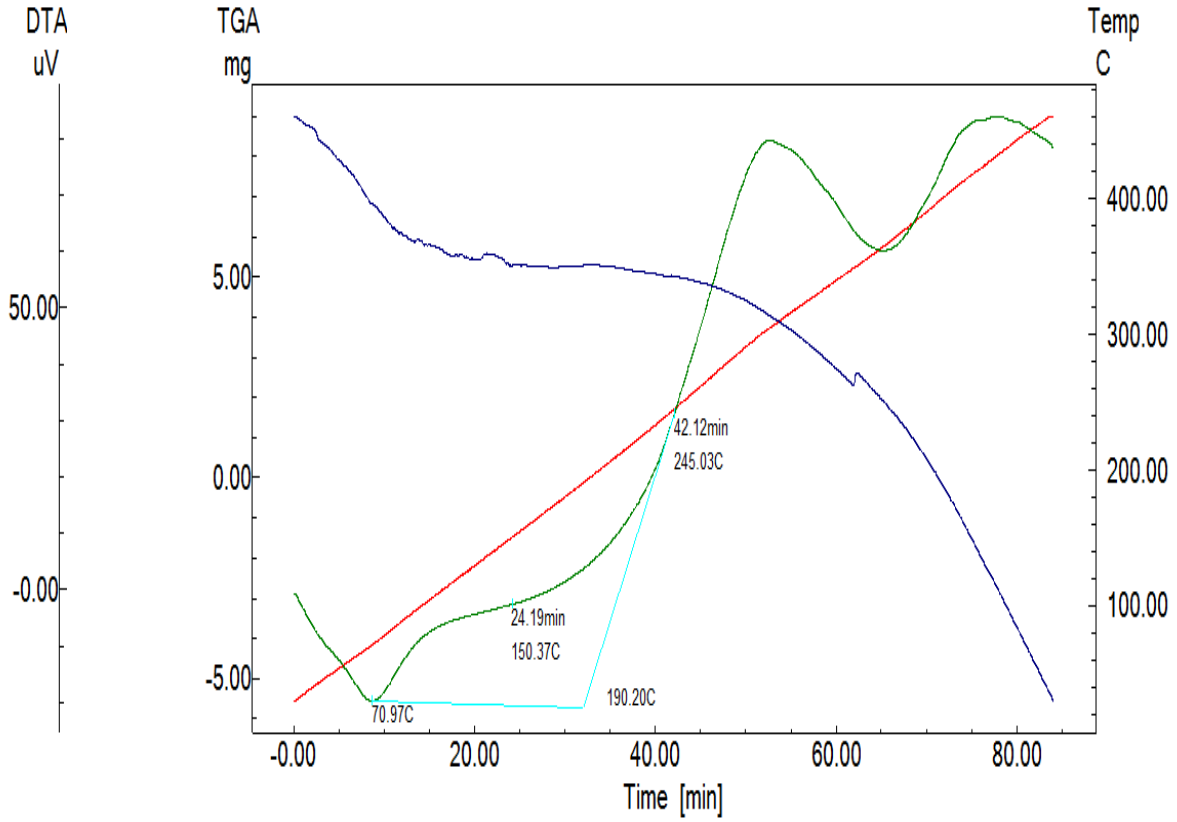

Figure A1-24:DTA Thermogram For MCL-1

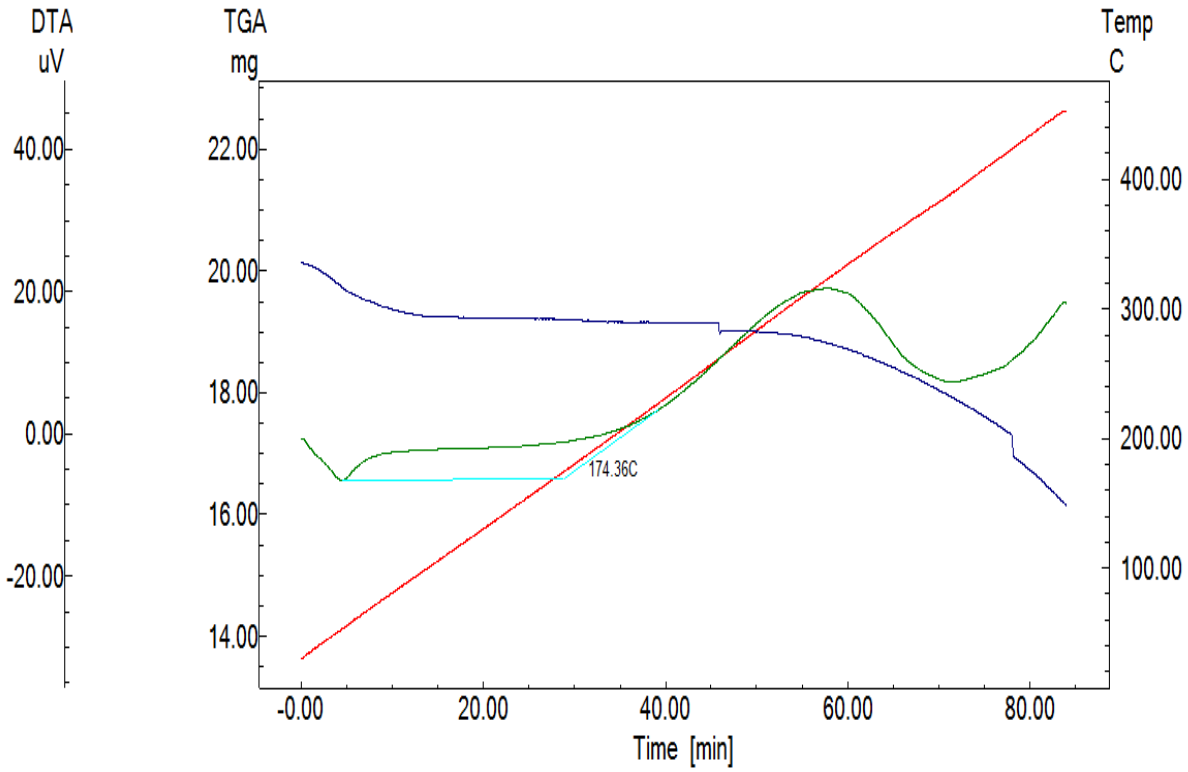

Figure A1-25: DTA Thermogram For MCL-2

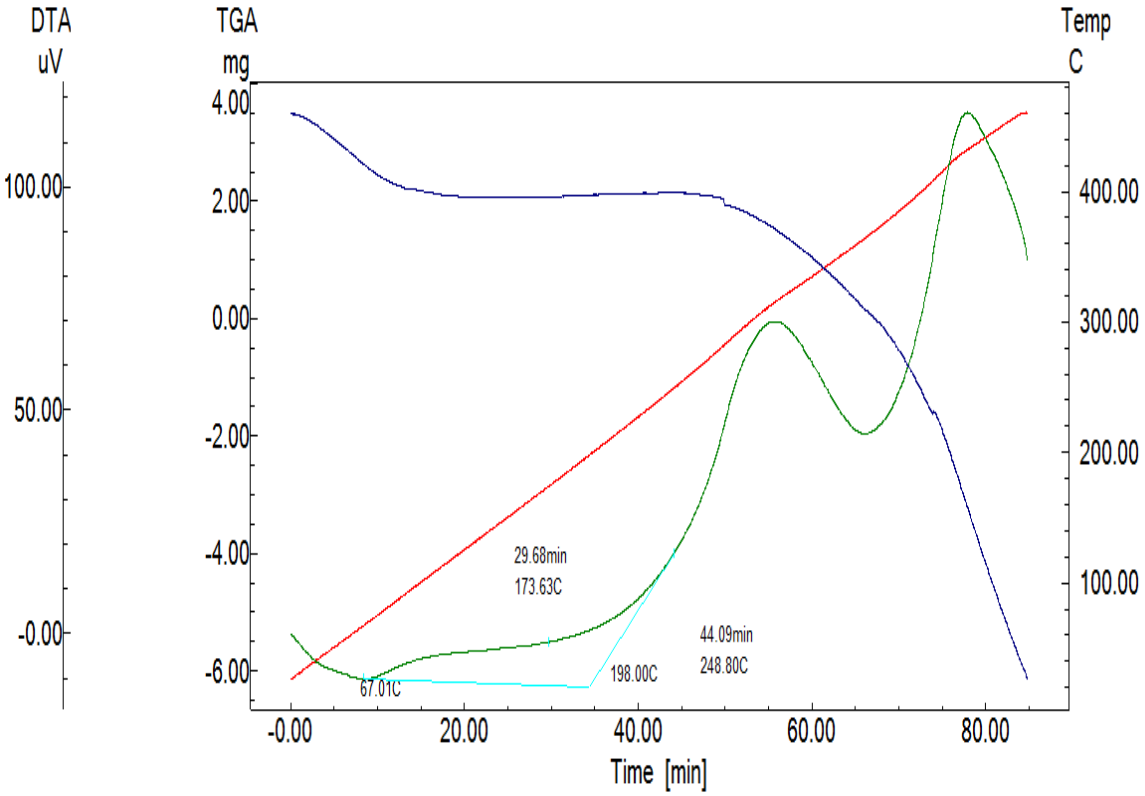

Figure A1-26:DTA Thermogram For MCL-3

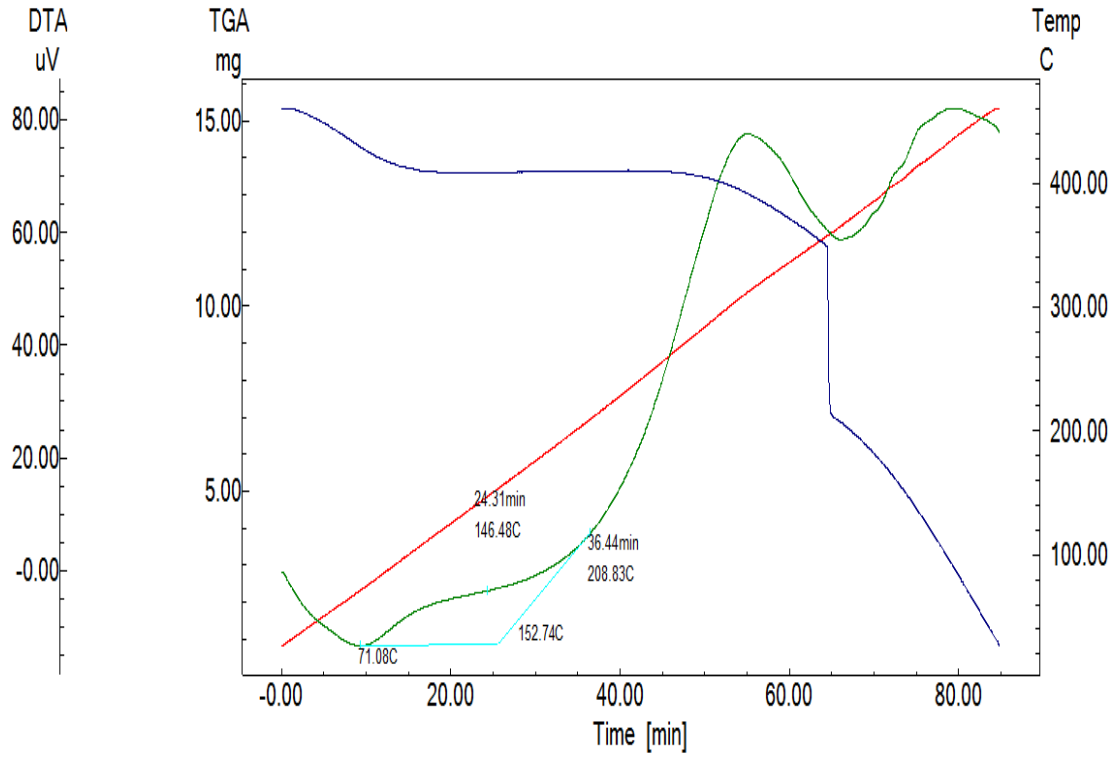

Figure A1-27: DTA Thermogram For MCL-4

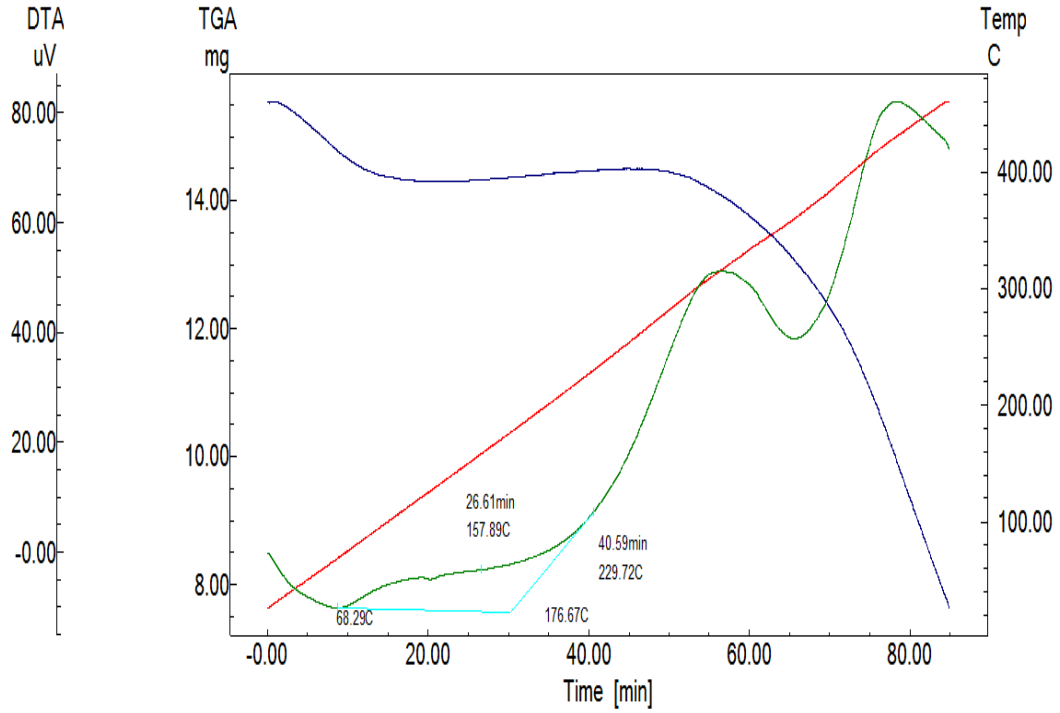

Figure A1-28: DTA Thermogram For MCL-5

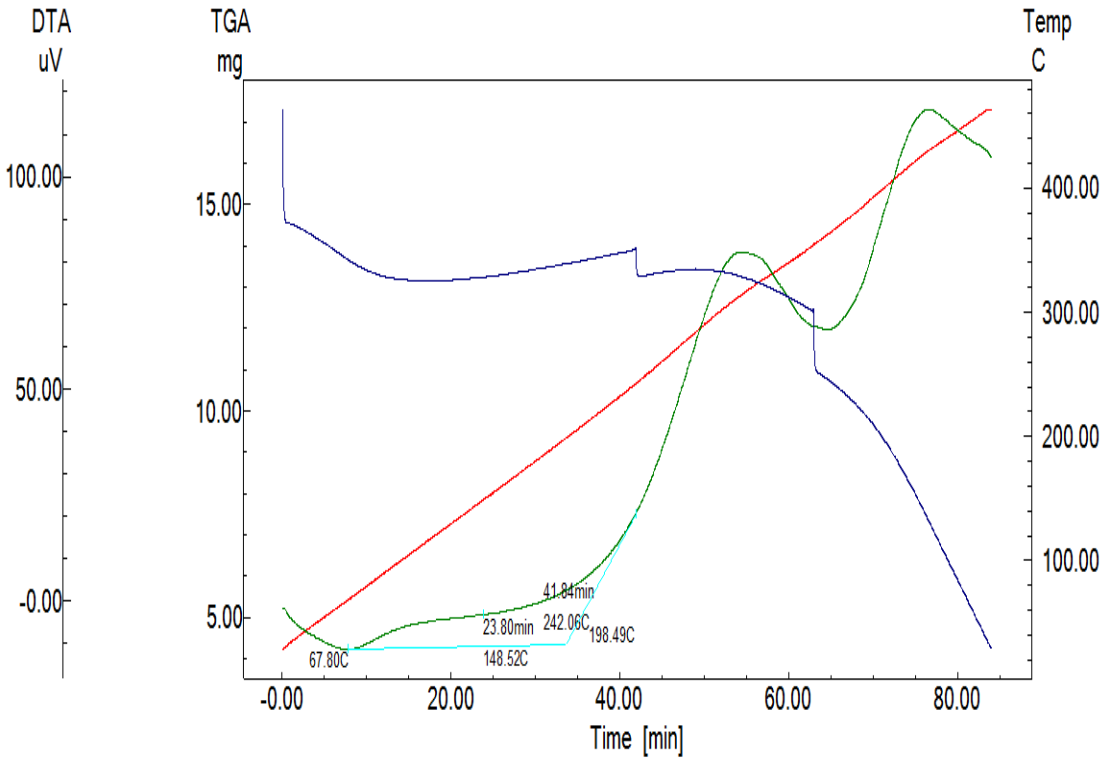

Figure A1-29: DTA Thermogram For MCL-6

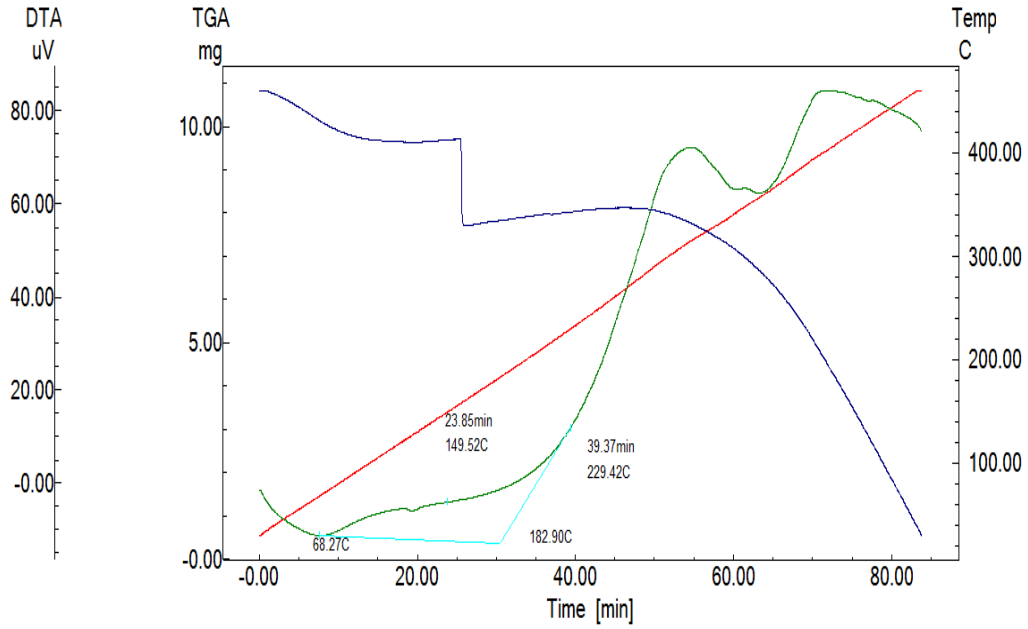

Figure A1-30:DTA Thermogram For MCL-7

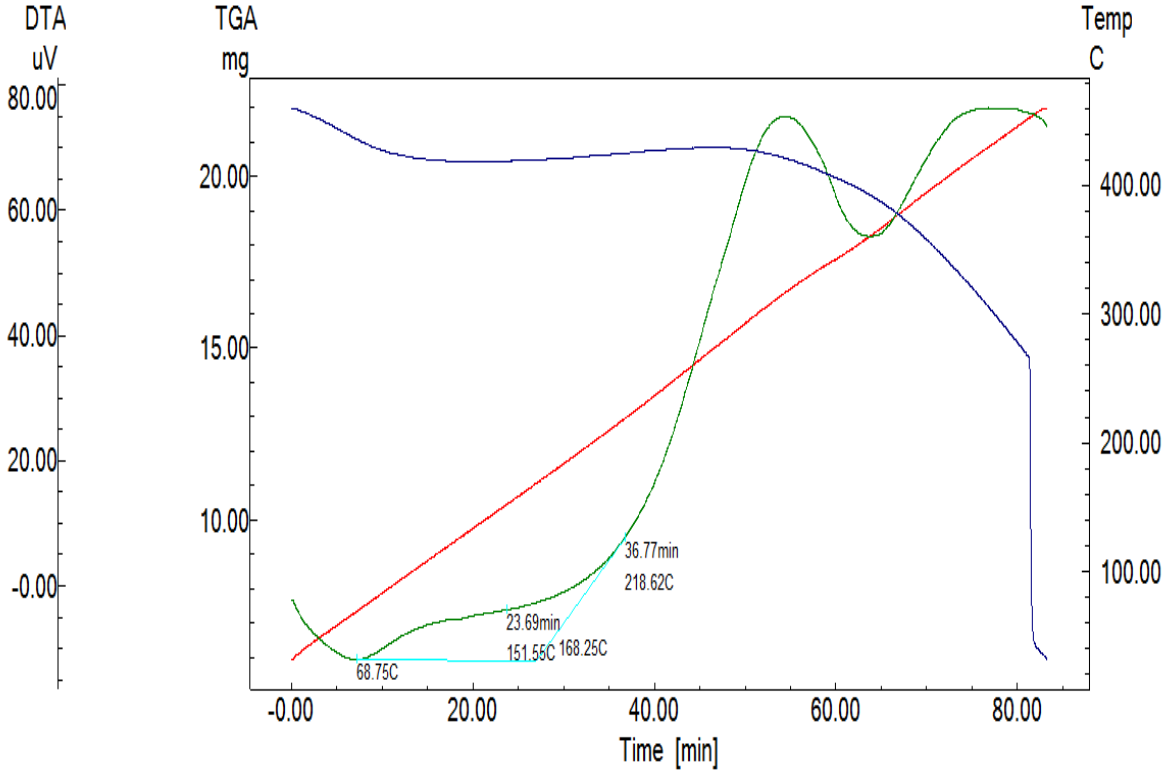

Figure A1-31: DTA Thermogram For MCL-8

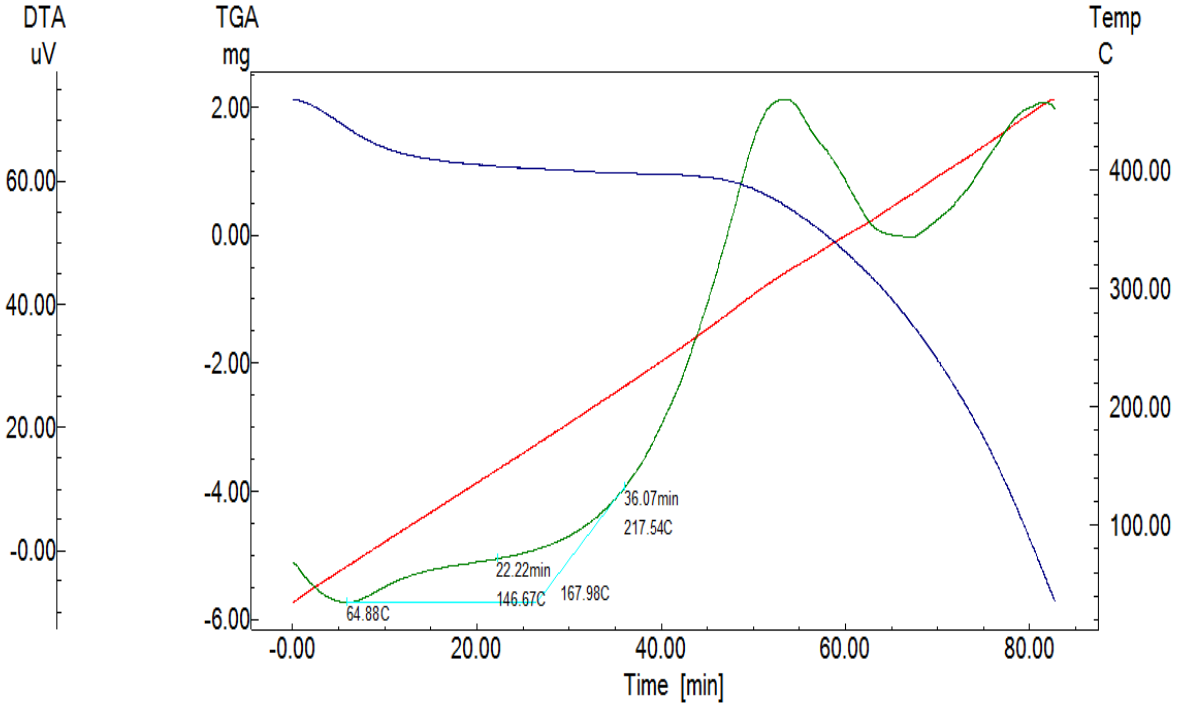

Figure A1-32: DTA Thermogram For MCL-9

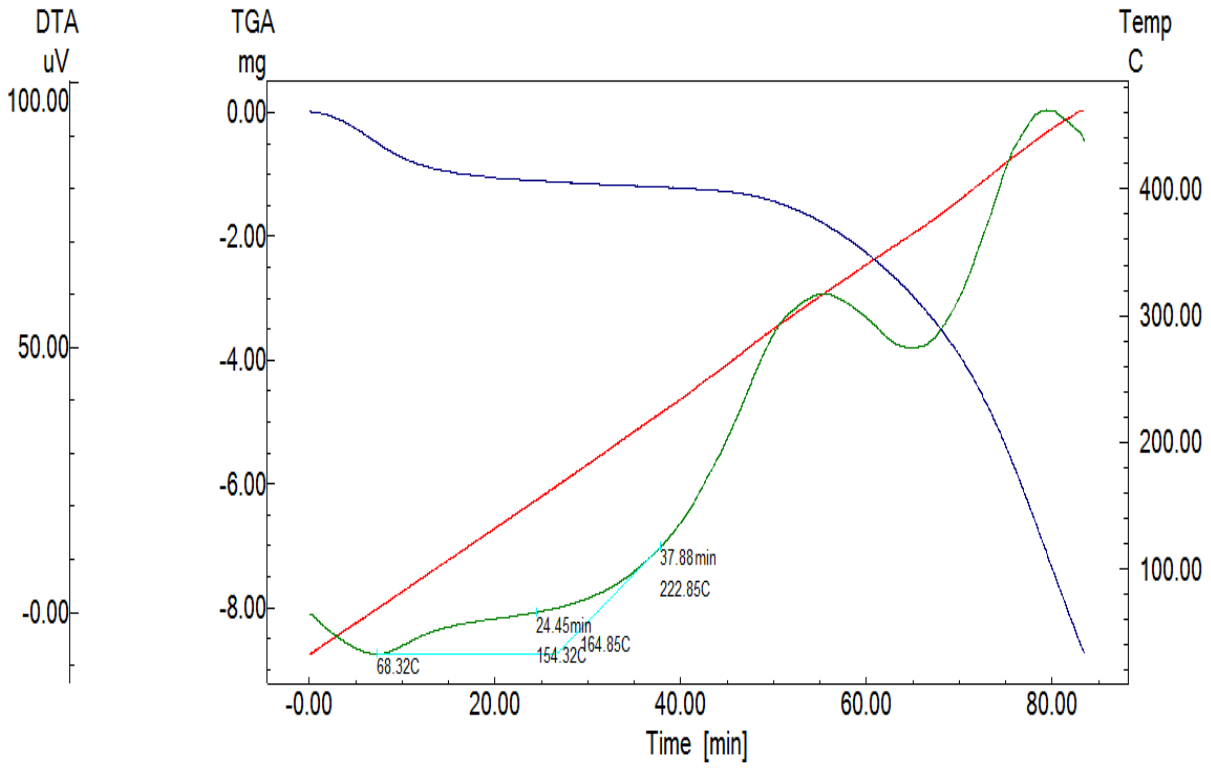

Figure A1-33: DTA Thermogram For MCL-10

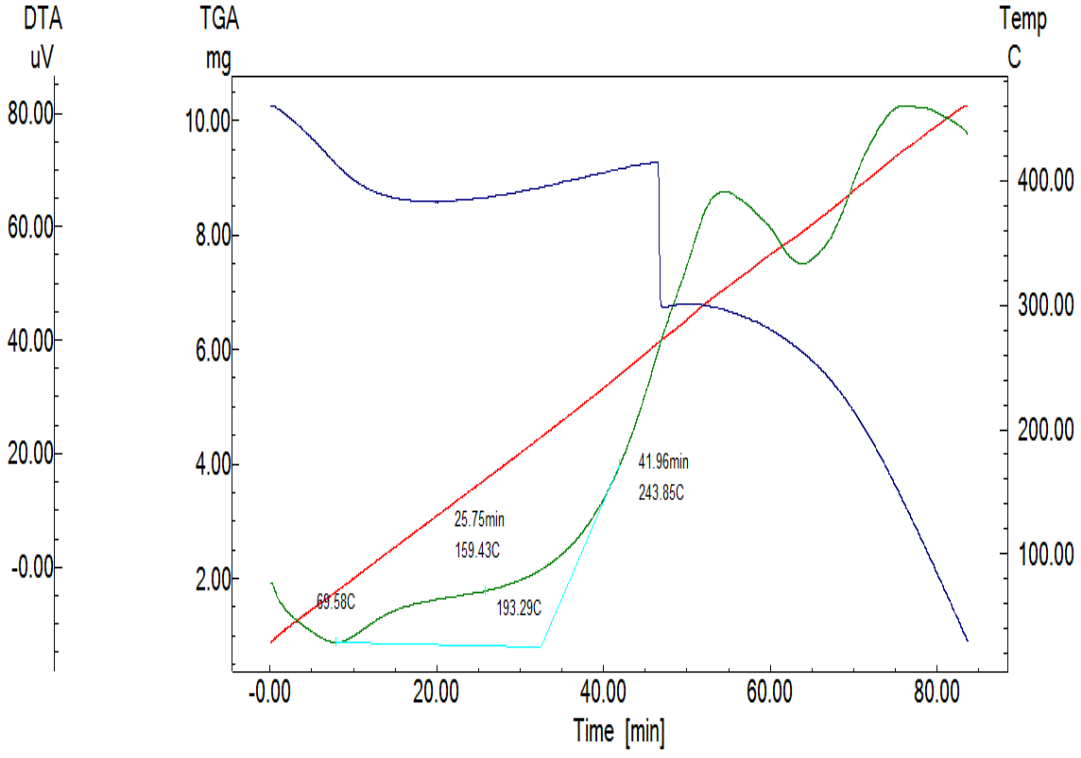

Figure A1-34:DTA Thermogram For MCL-11

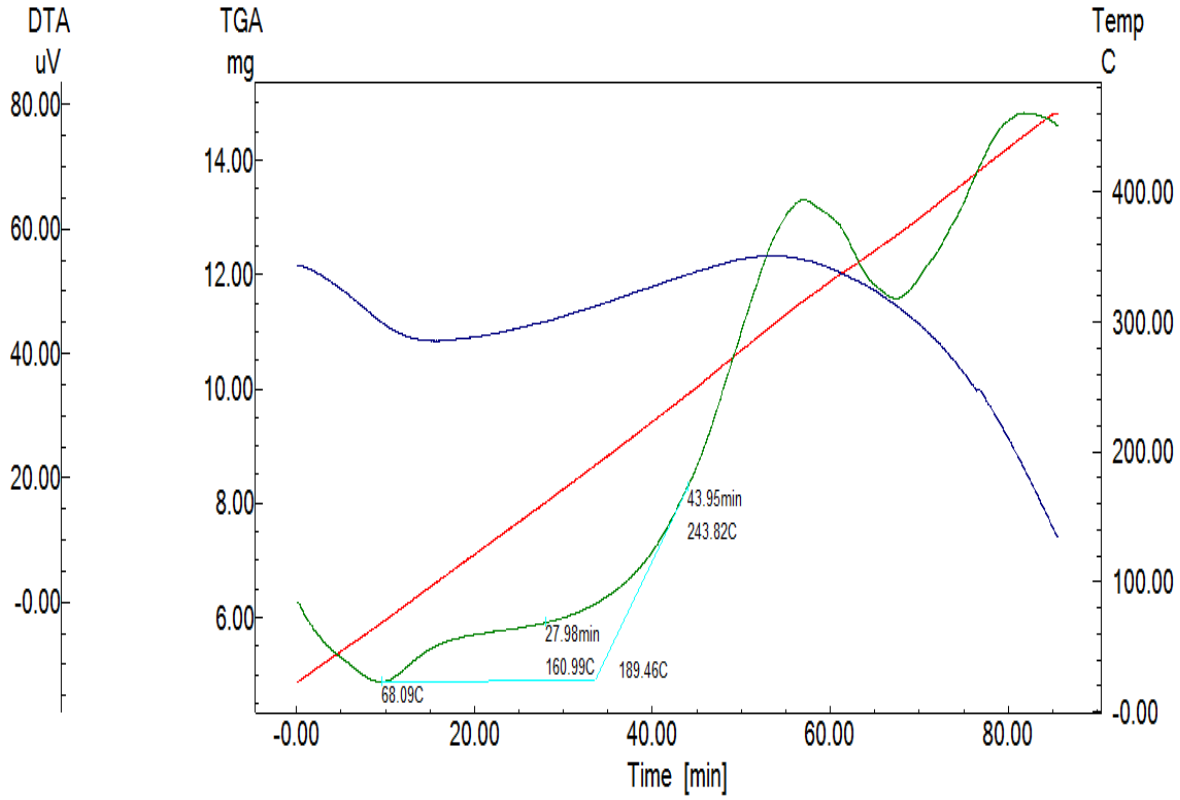

Figure A1-35: DTA Thermogram For MCL-12

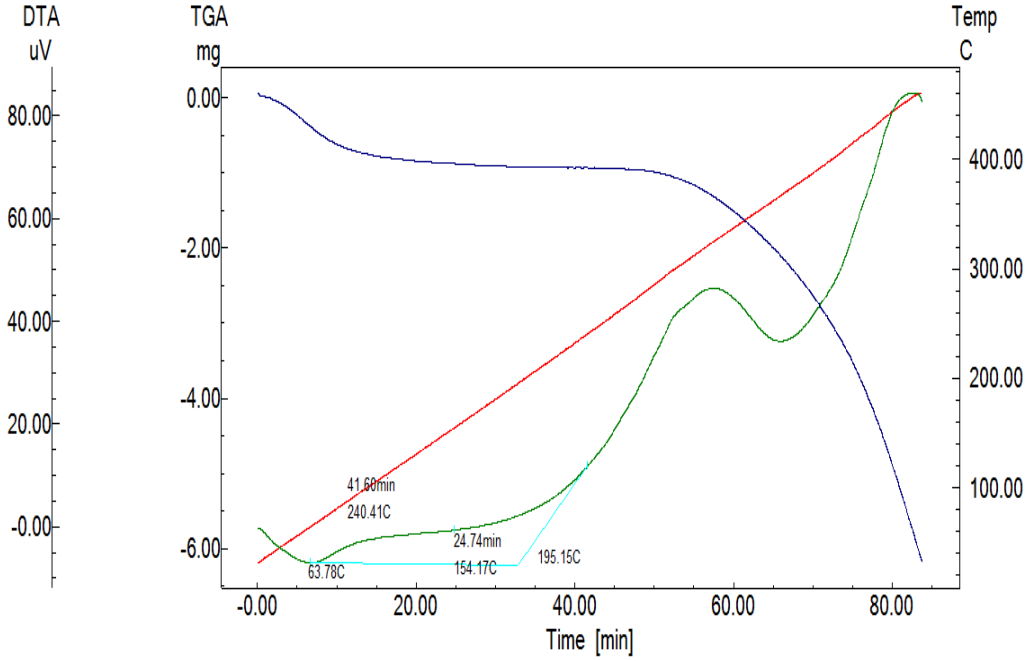

Figure A1-36:DTA Thermogram For MCL-13

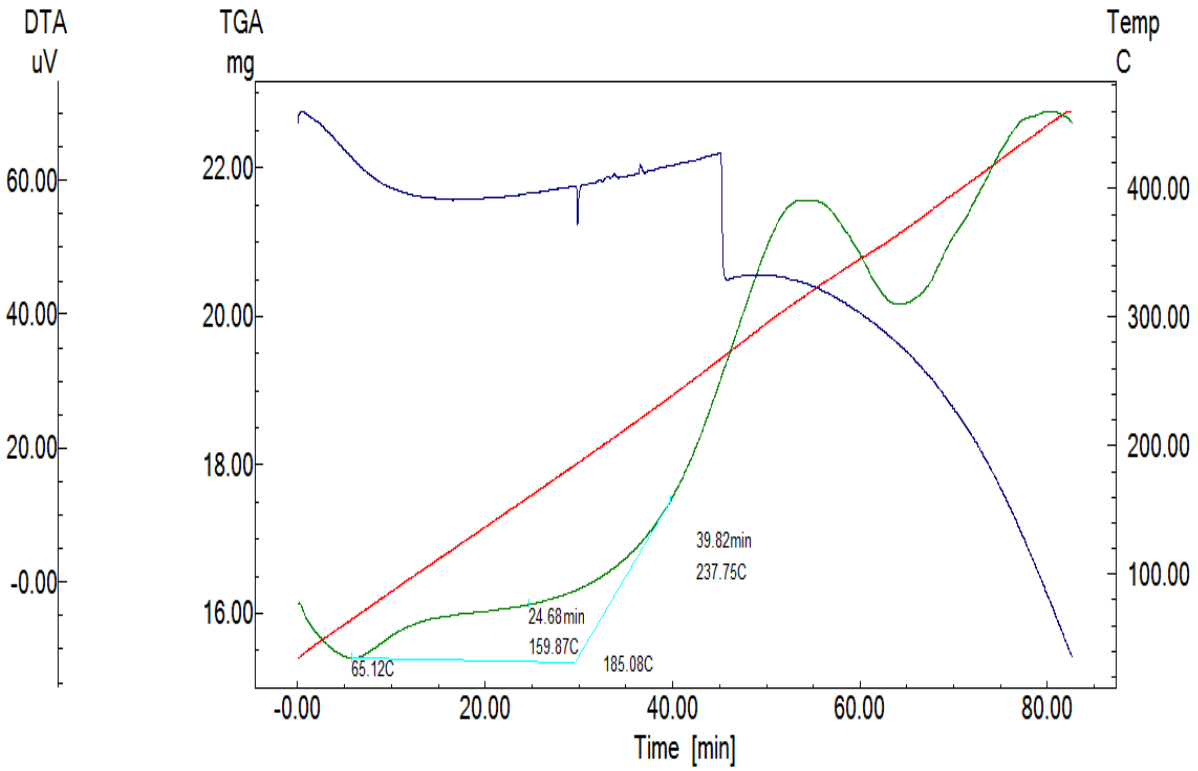

Figure A1-37: DTA Thermogram For MCL-14

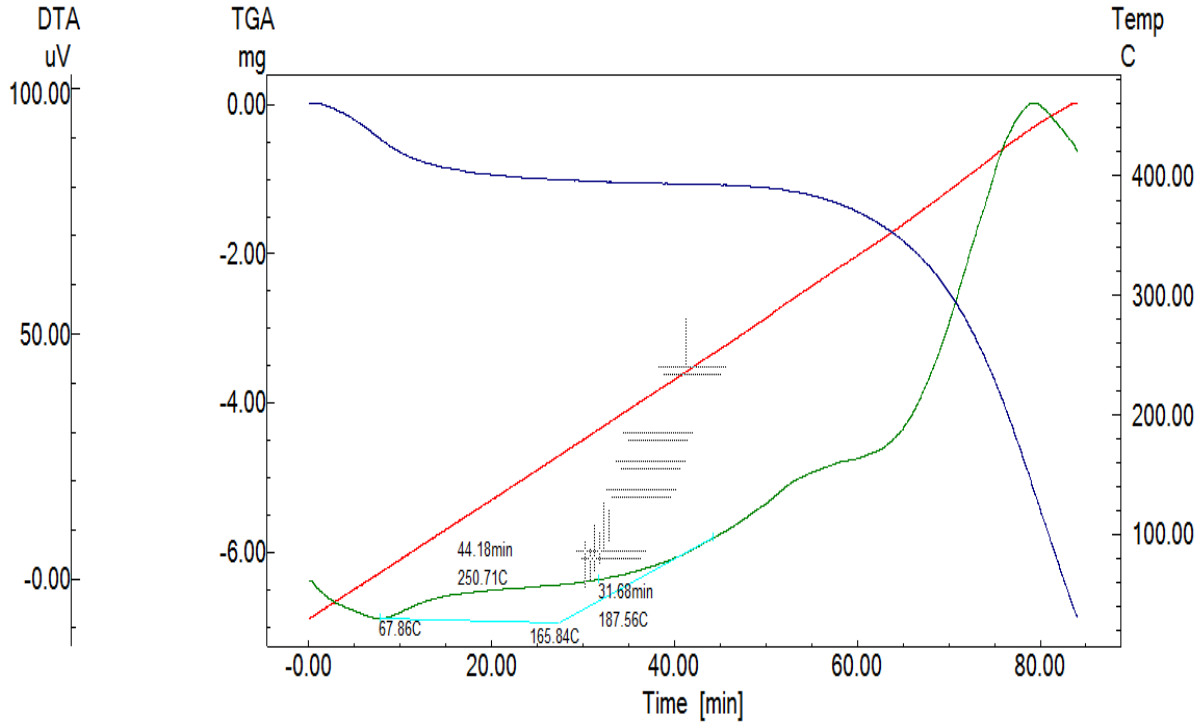

Figure A1-38: DTA Thermogram For MCL-15

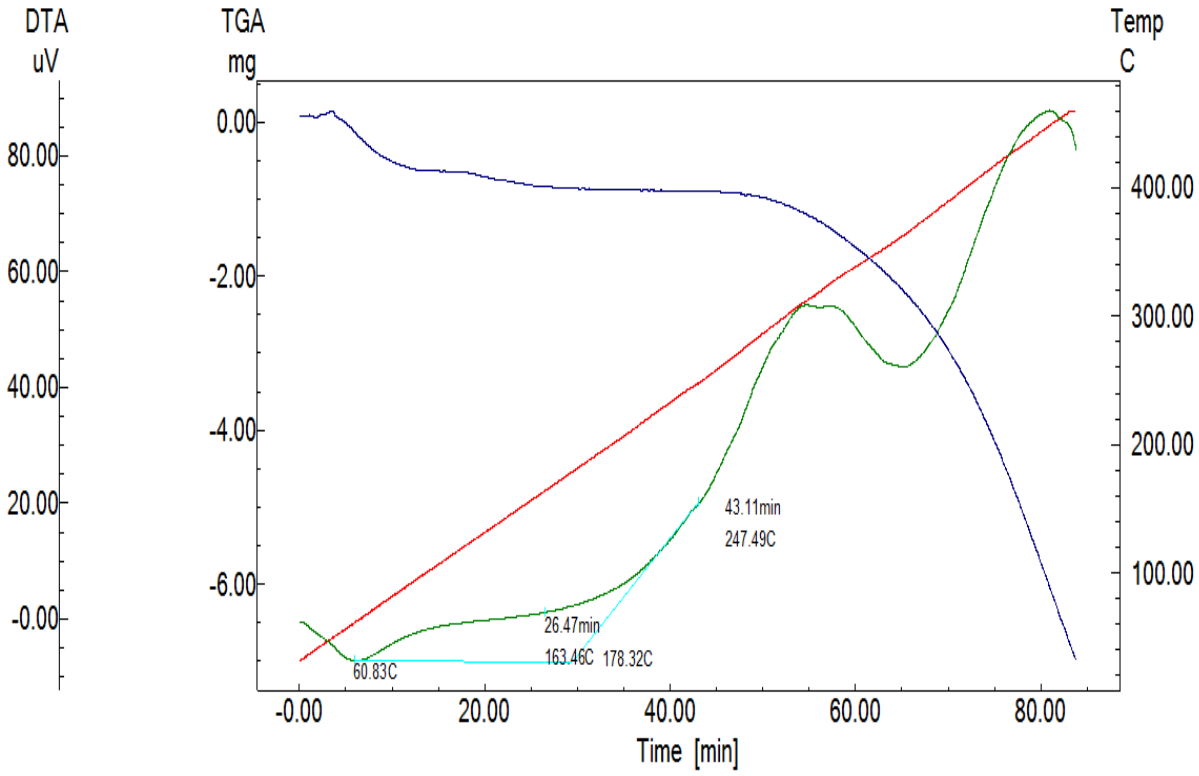

Figure A1-39: DTA Thermogram for MCL-16

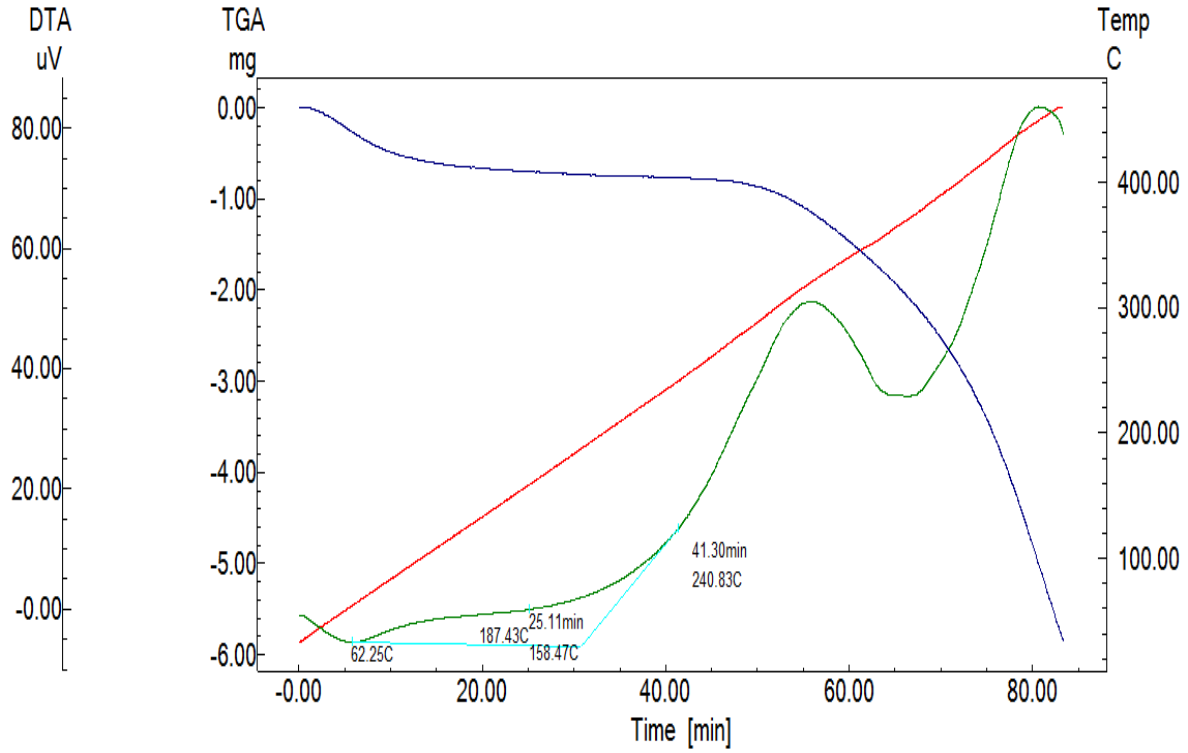

Figure A1-40: DTA Thermogram for MCL-17

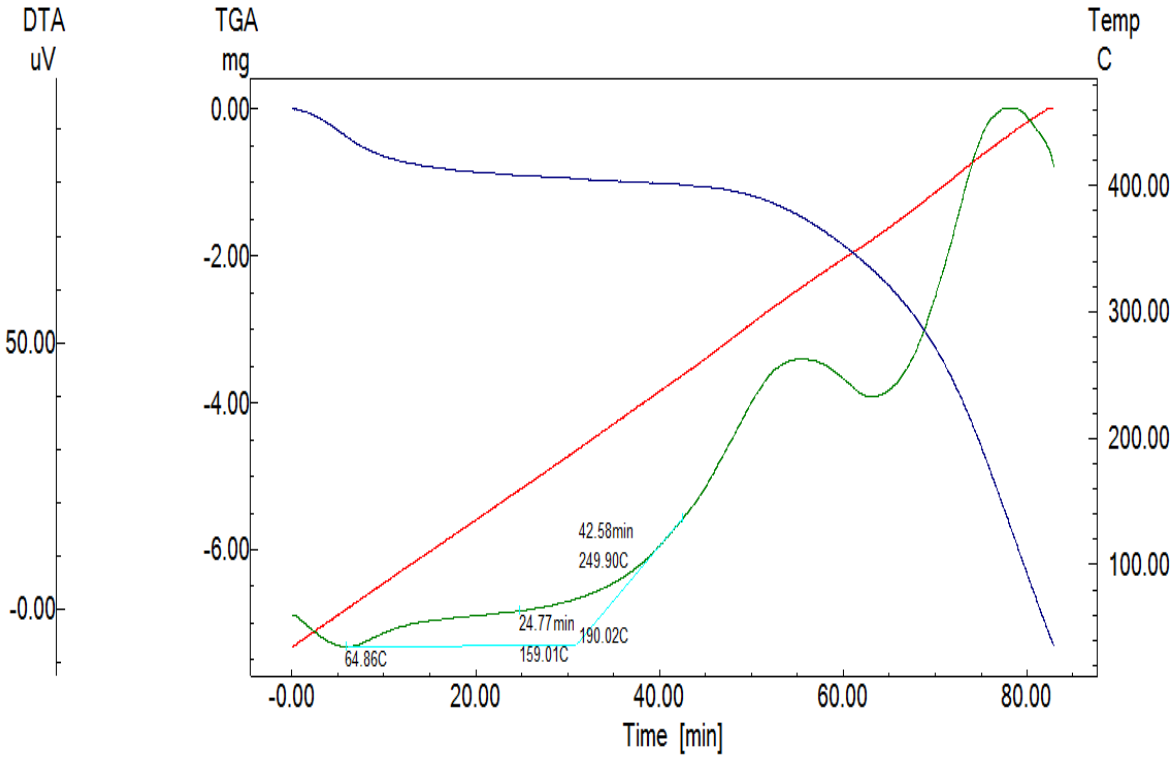

Figure A1-41: DTA Thermogram for MCL-18

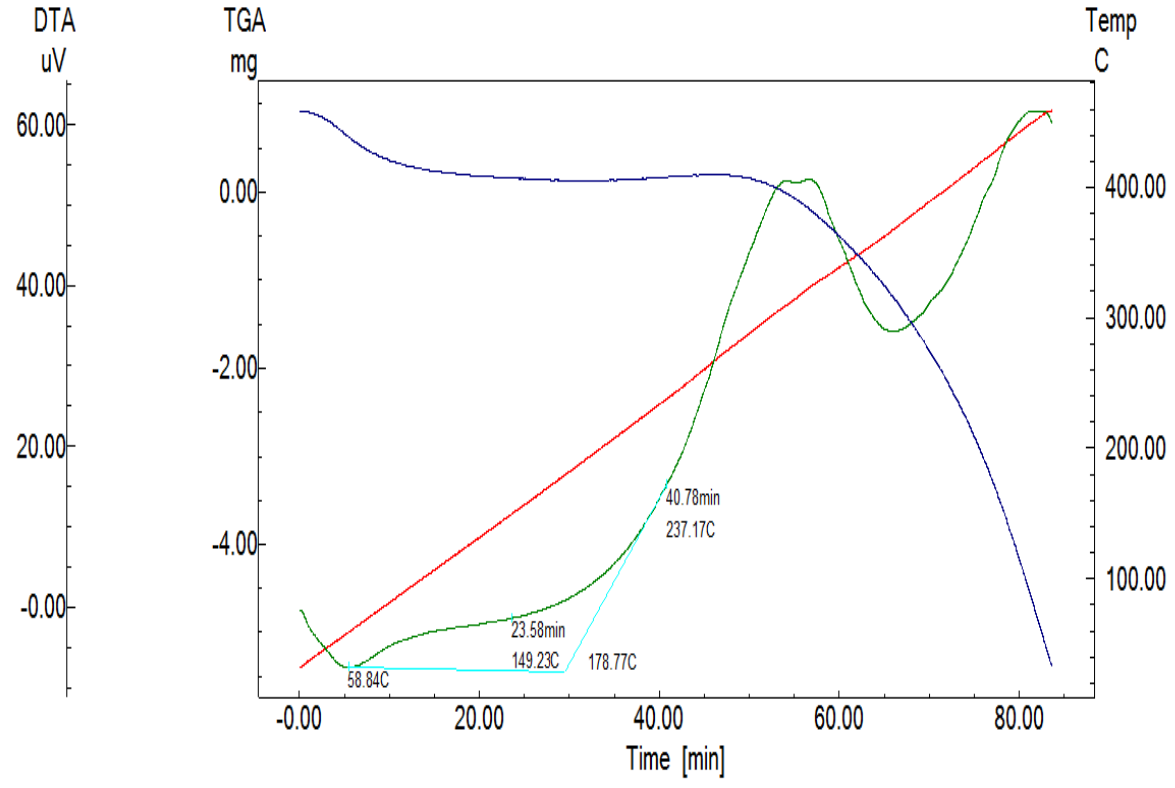

Figure A1-42: DTA Thermogram for MCL-19

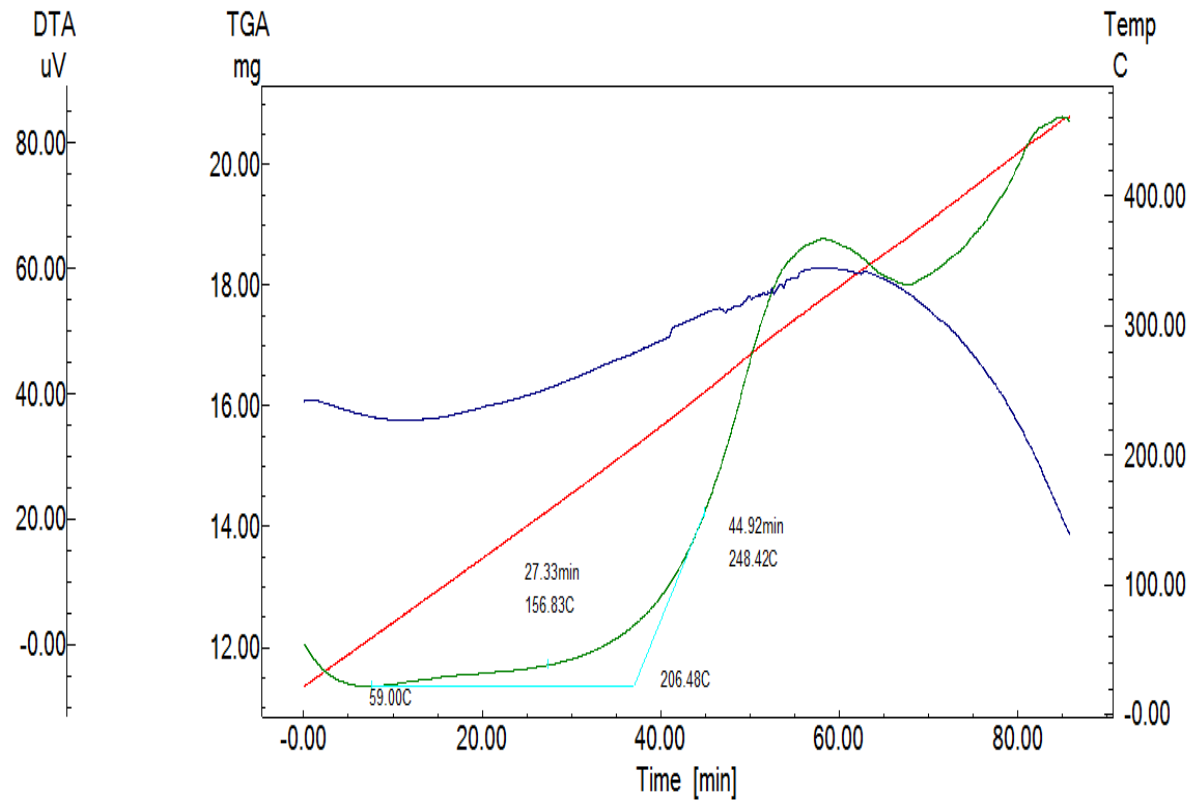

Figure A1-43: DTA Thermogram for BCCL-1

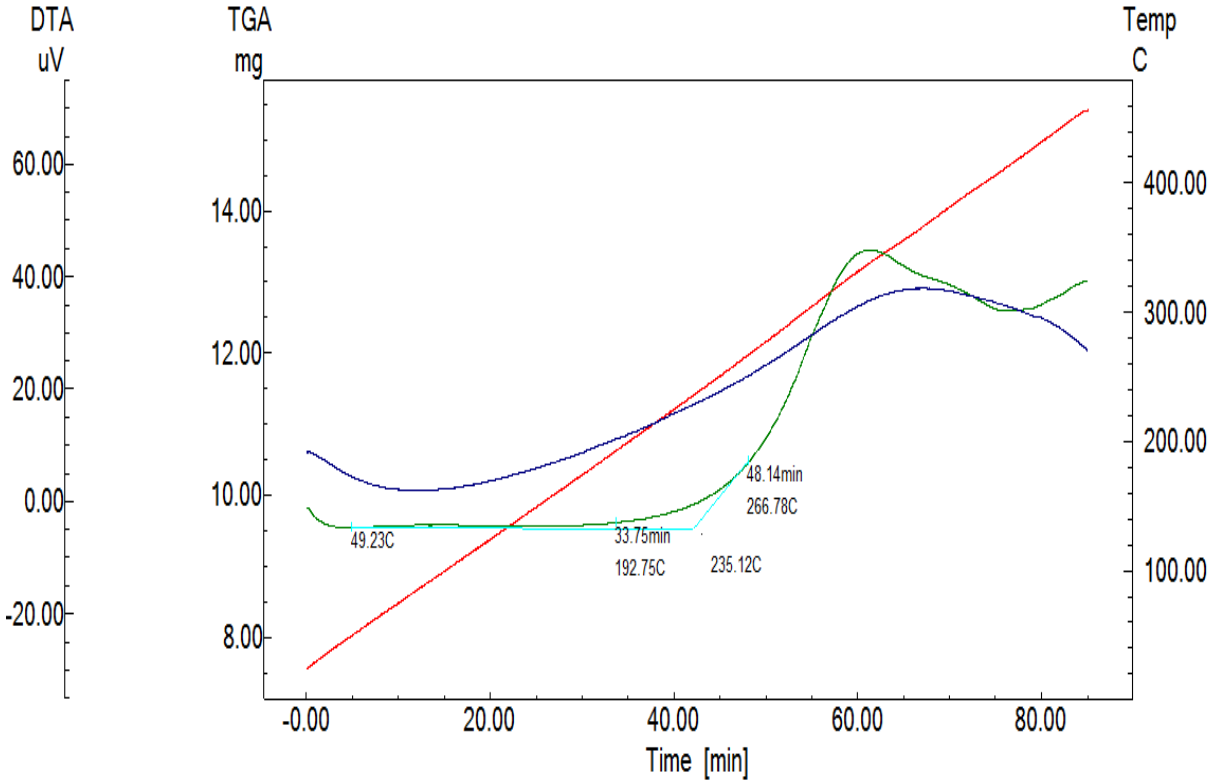

Figure A1-44: DTA Thermogram for BCCL-3

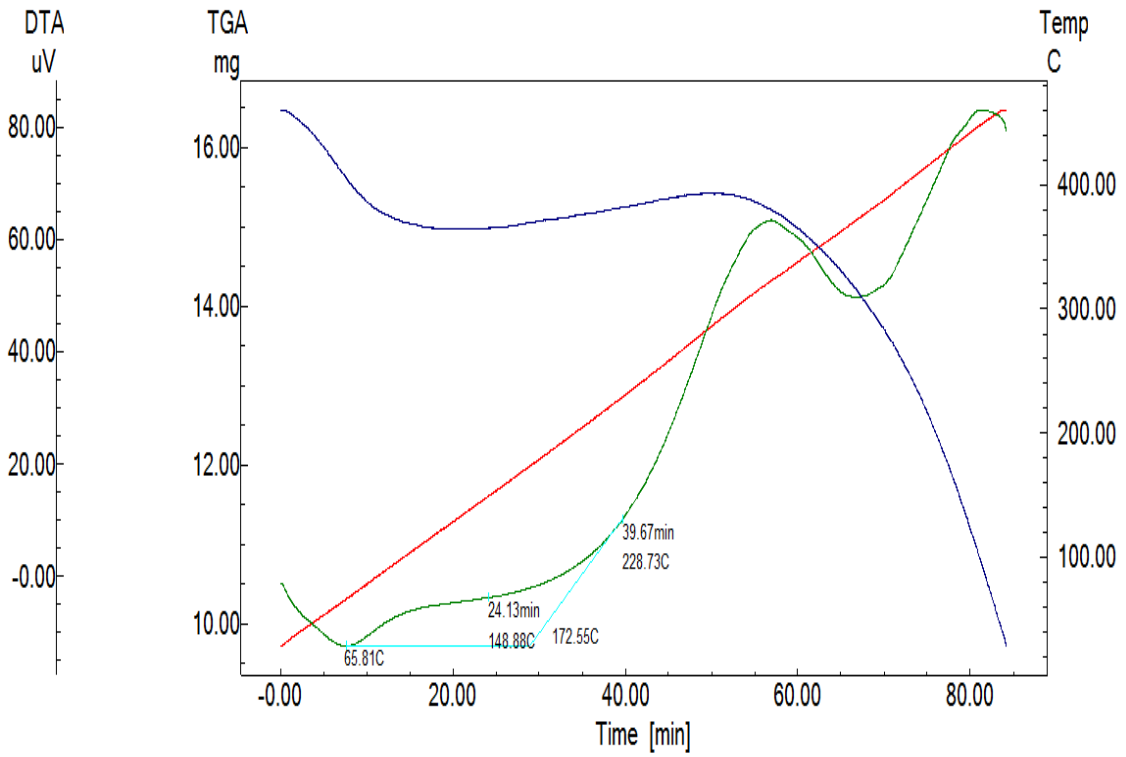

Figure A1-45: DTA Thermogram for WCL-1

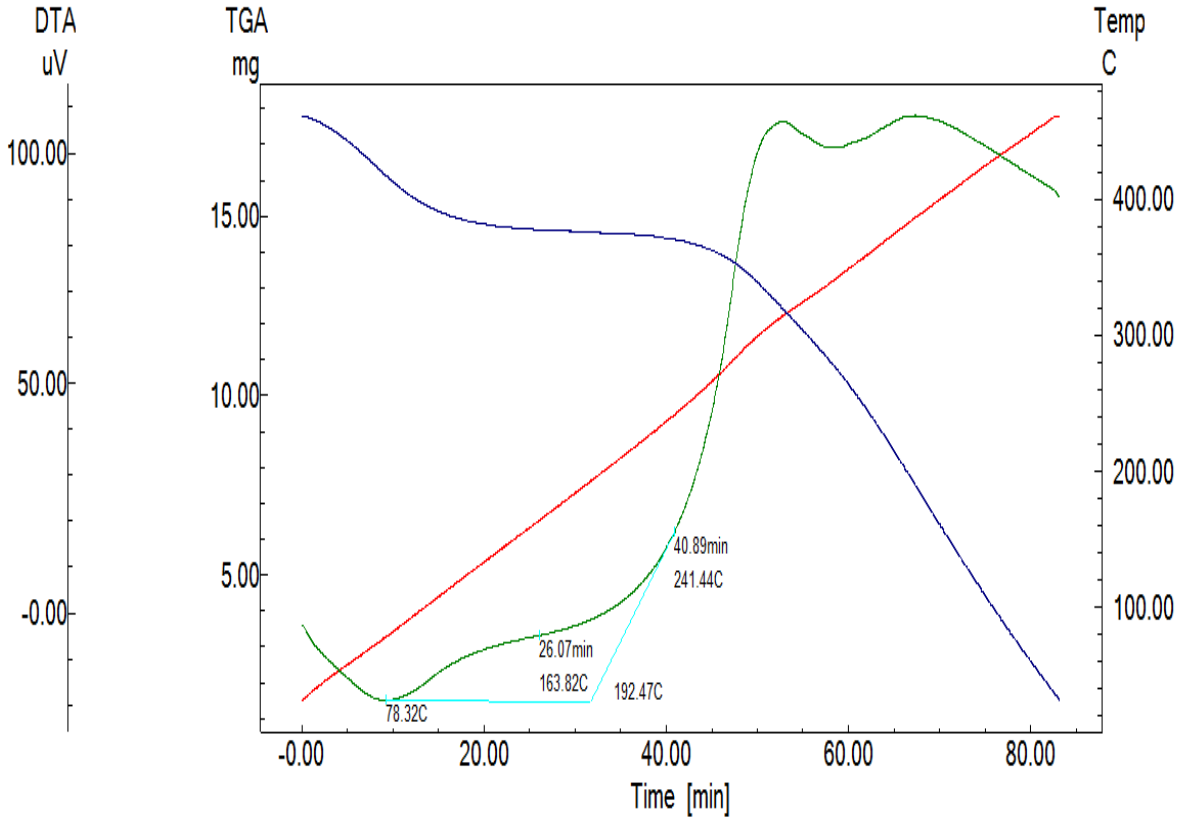

Figure A1-46: DTA Thermogram for NLC-1

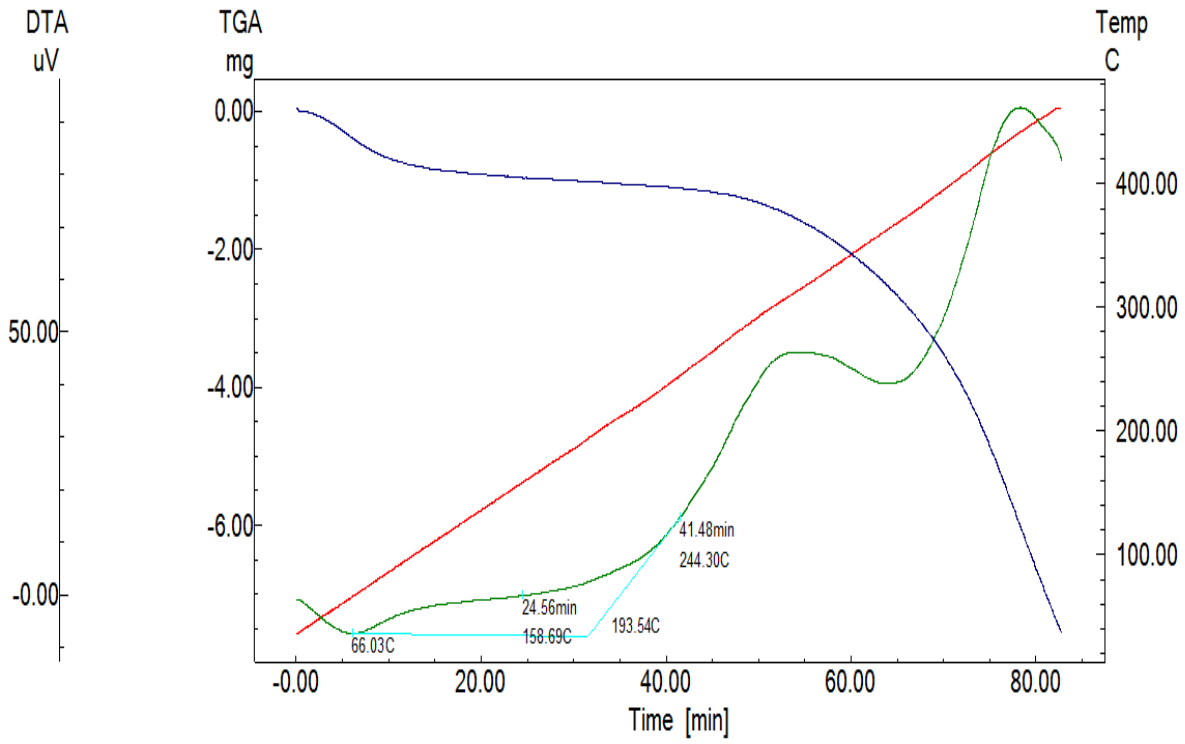

Figure A1-47: DTA Thermogram for NCL-1

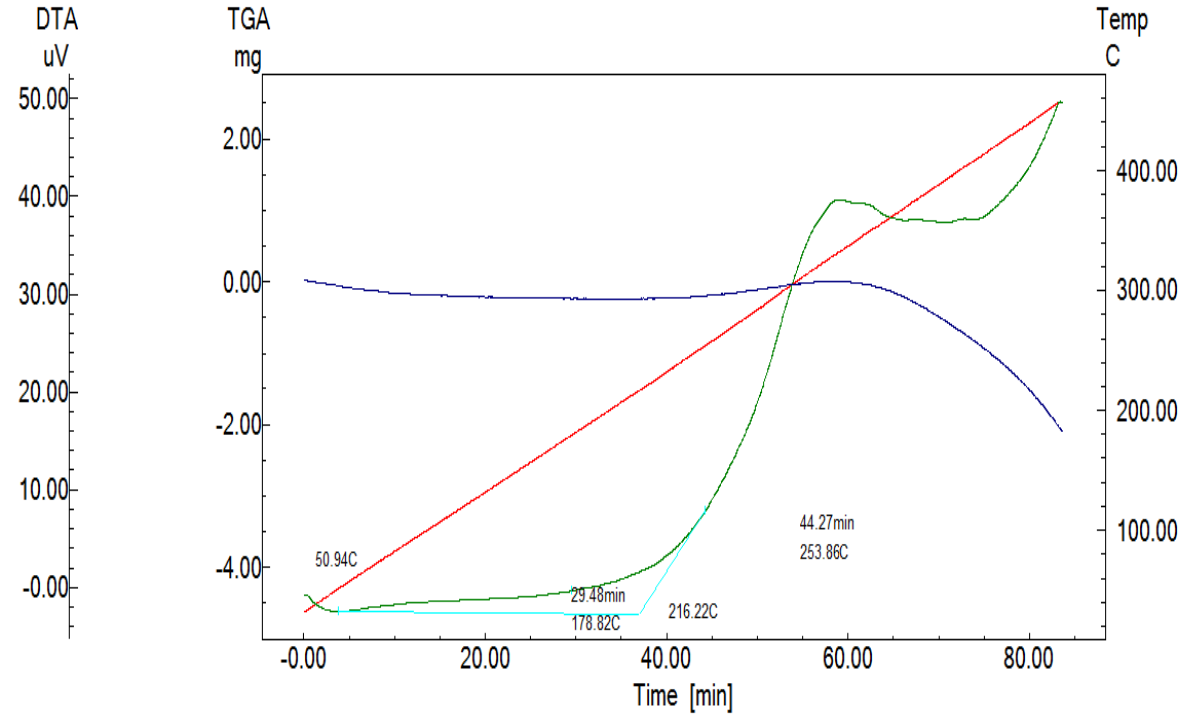

Figure A1-48: DTA Thermogram for AUSTRALIAN COAL (NSW)

APPENDIX-2

FTIR Curves for Different Samples

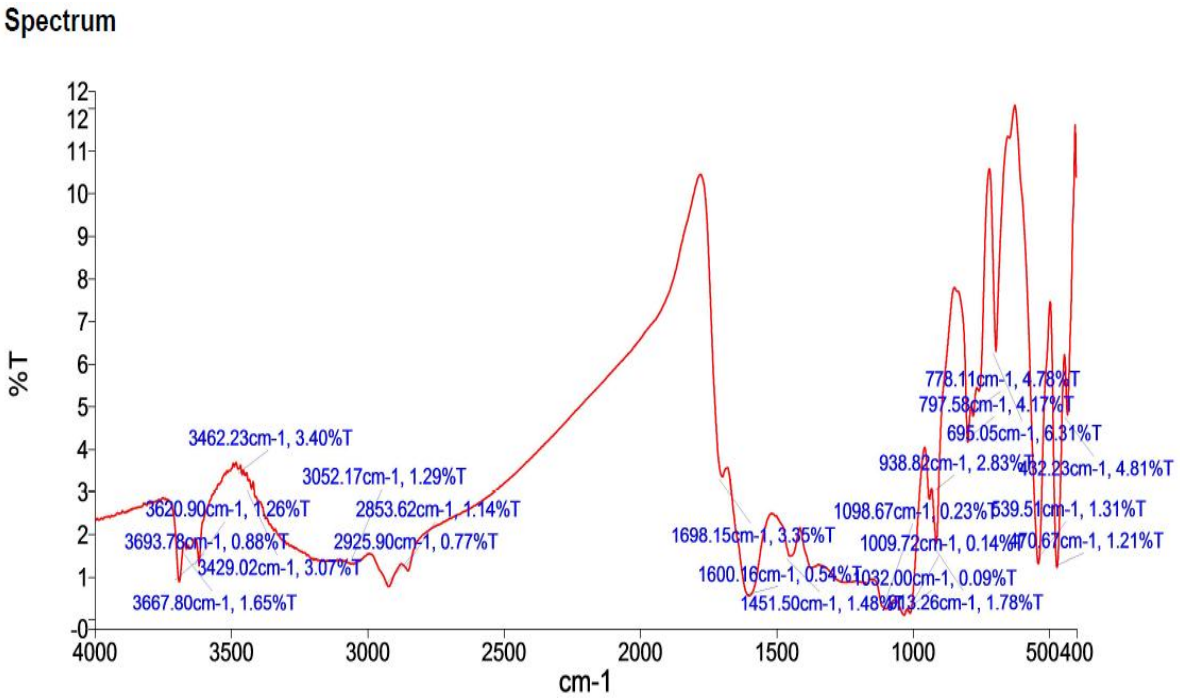

Figure A2-1: FTIR Transmittance spectra for SCCL-1

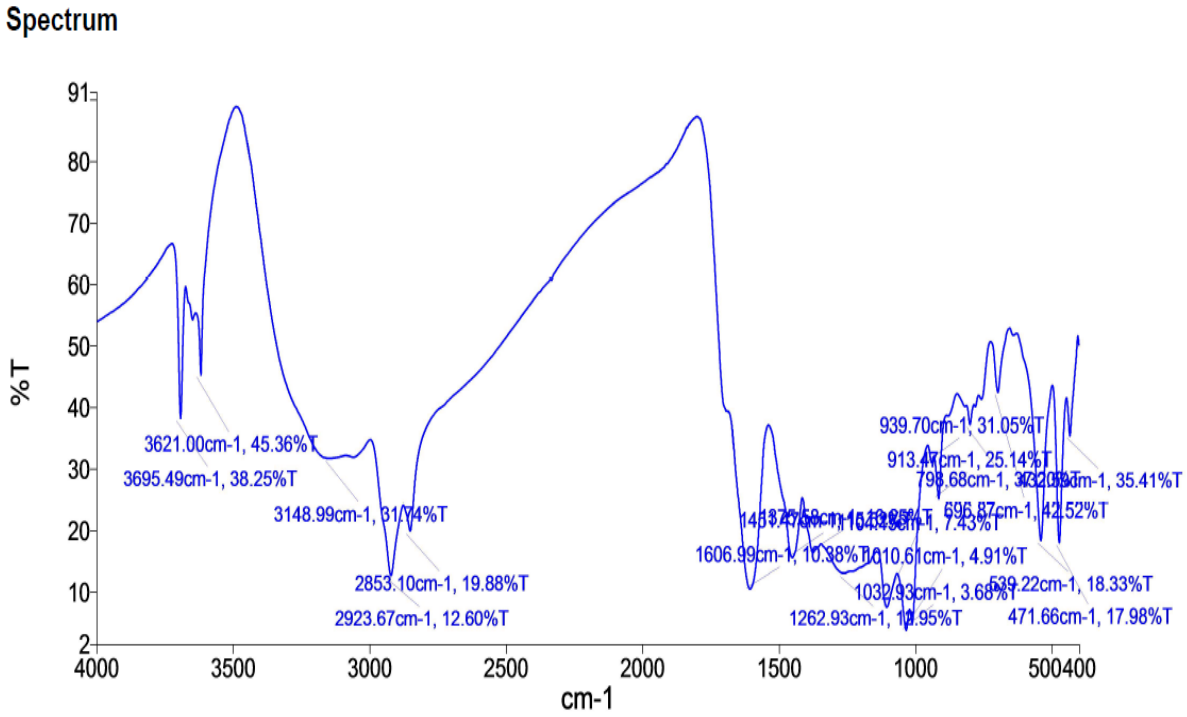

Figure A2-2: FTIR Transmittance spectra for SCCL-2

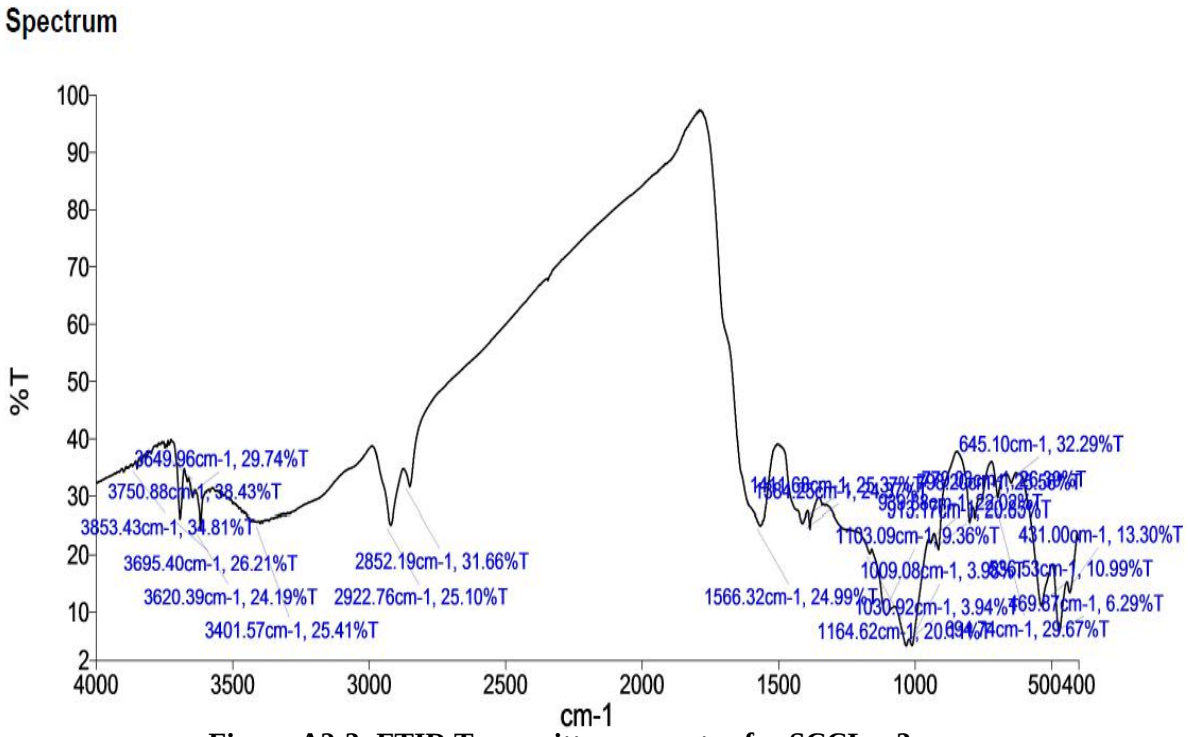

Figure A2-3: FTIR Transmittance spectra for SCCL – 3

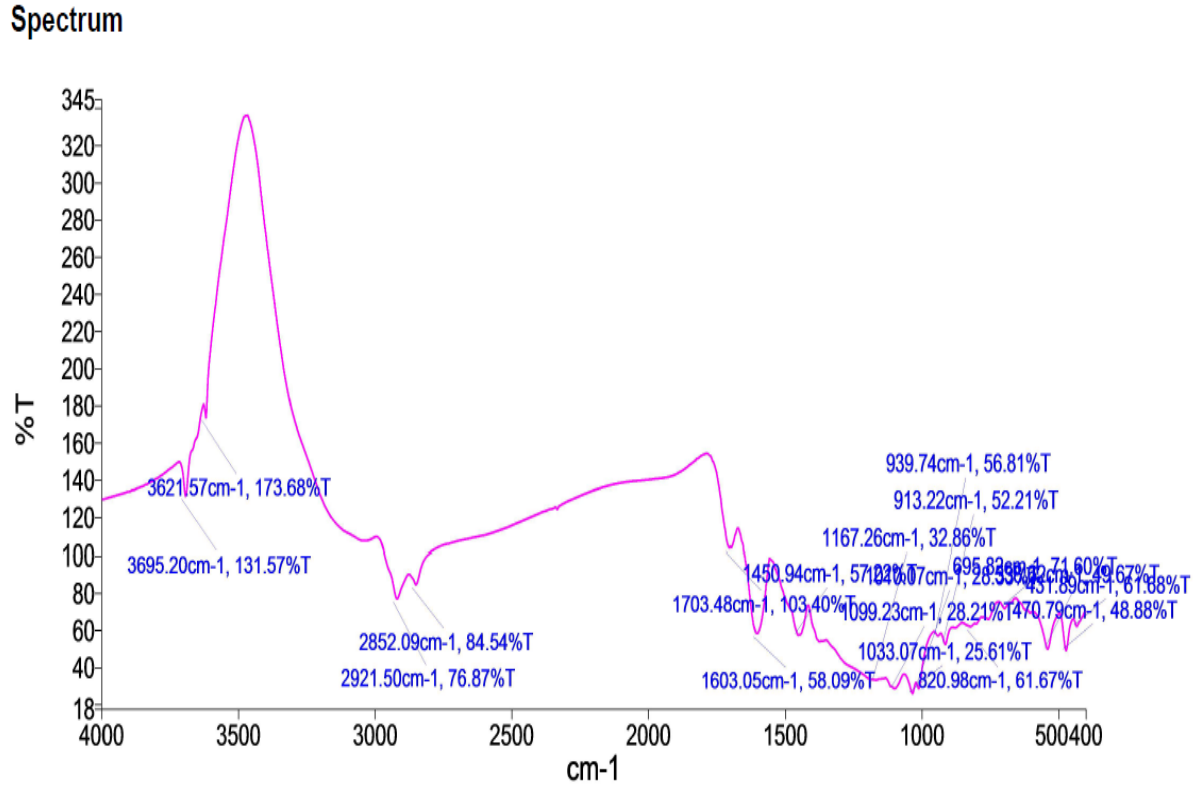

Figure A2-4: FTIR Transmittance spectra for SCCL-4

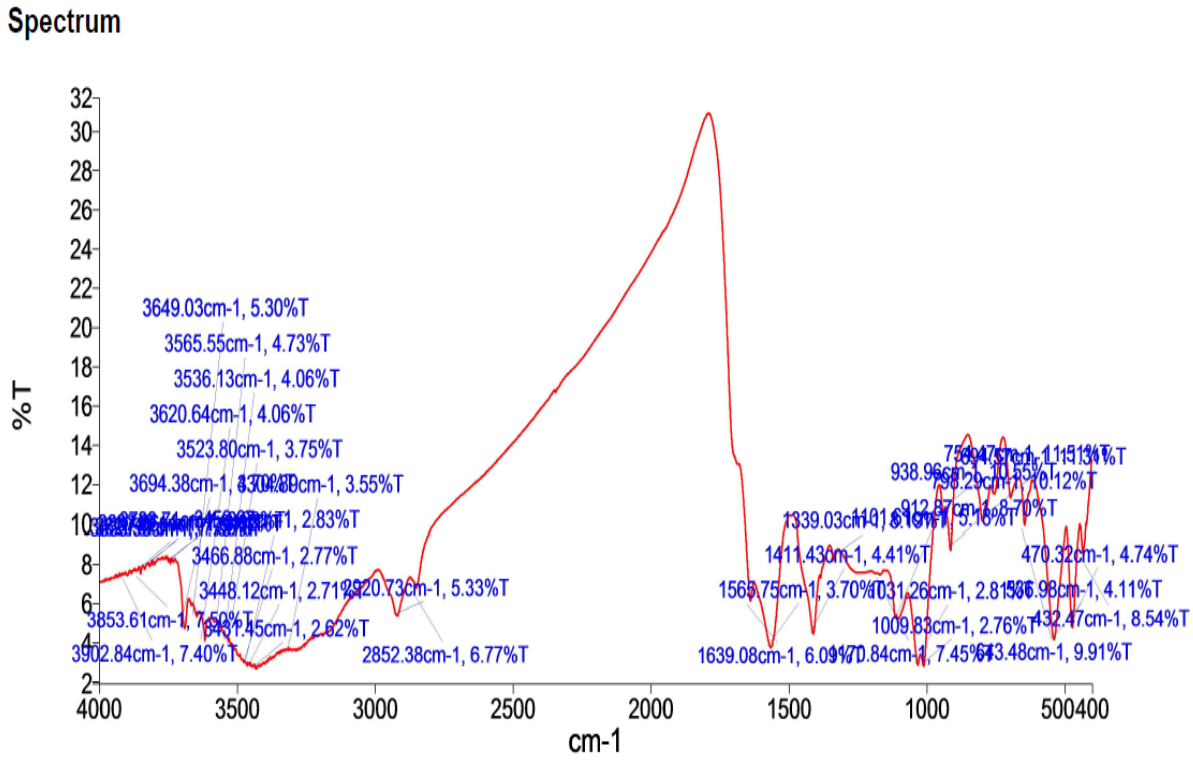

Figure A2-5: FTIR Transmittance spectra for SCCL – 5

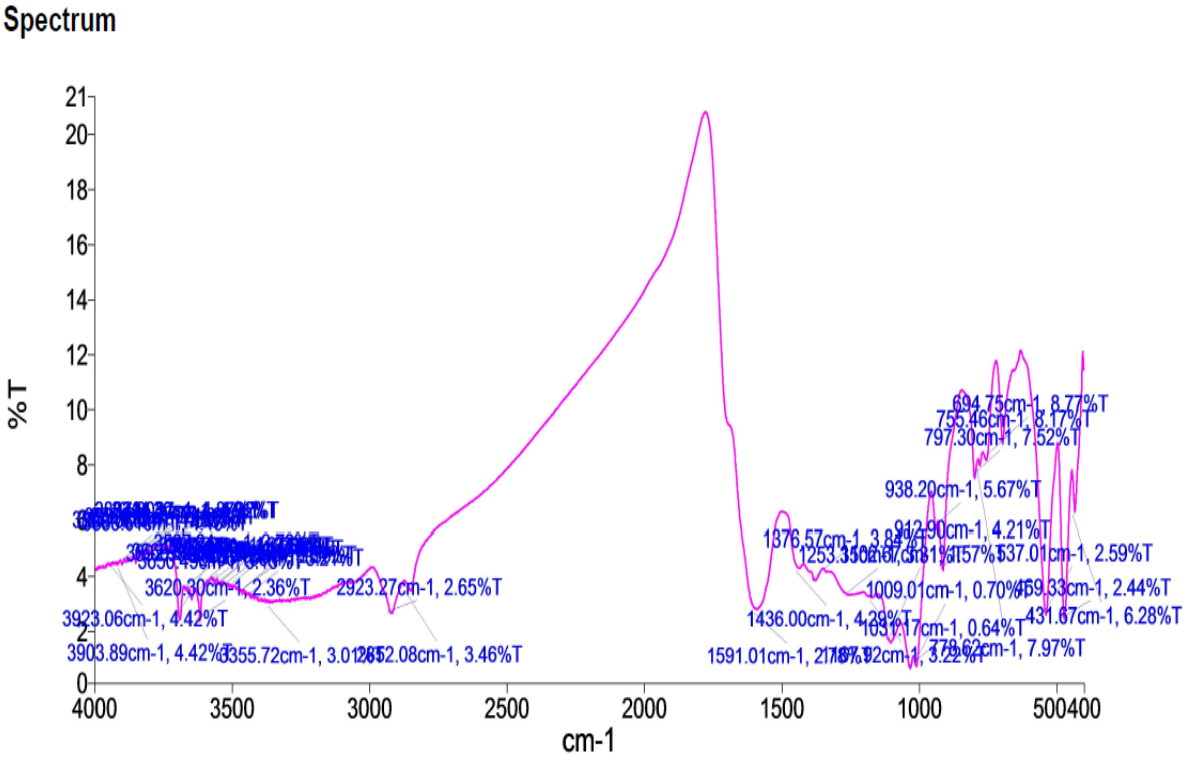

Figure A2-6: FTIR Transmittance spectra for SCCL-6

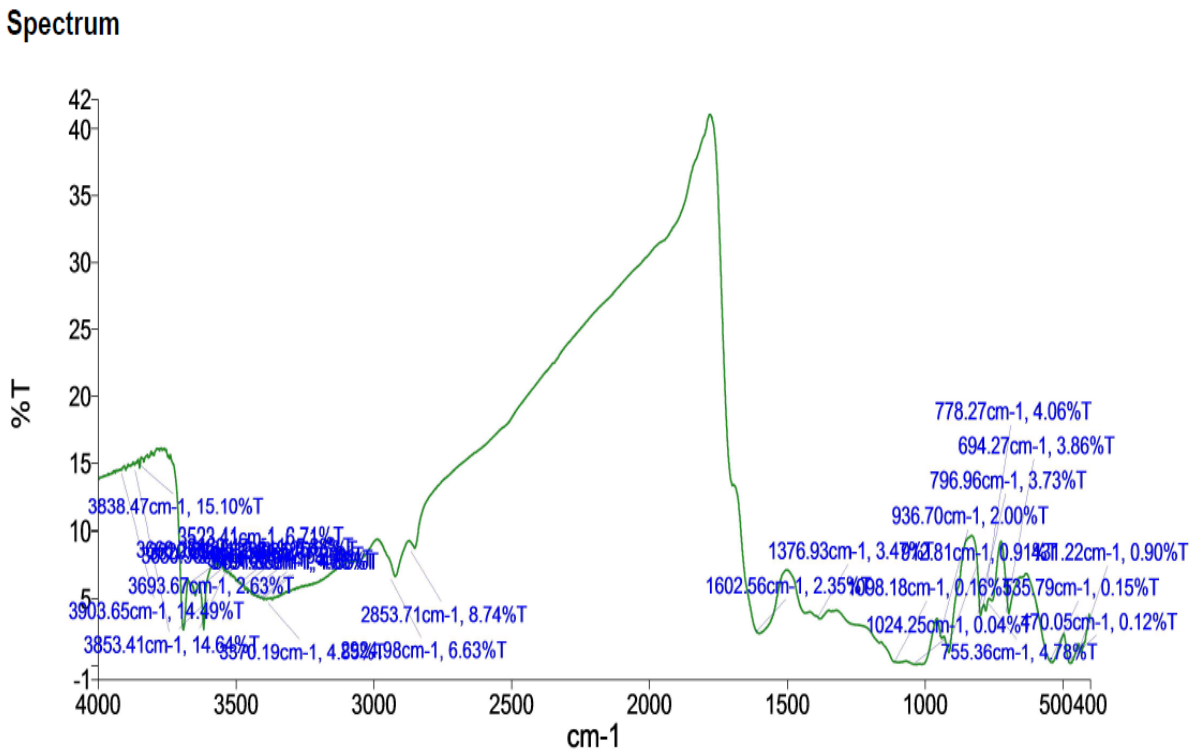

Figure A2-7: FTIR Transmittance spectra for SCCL-7

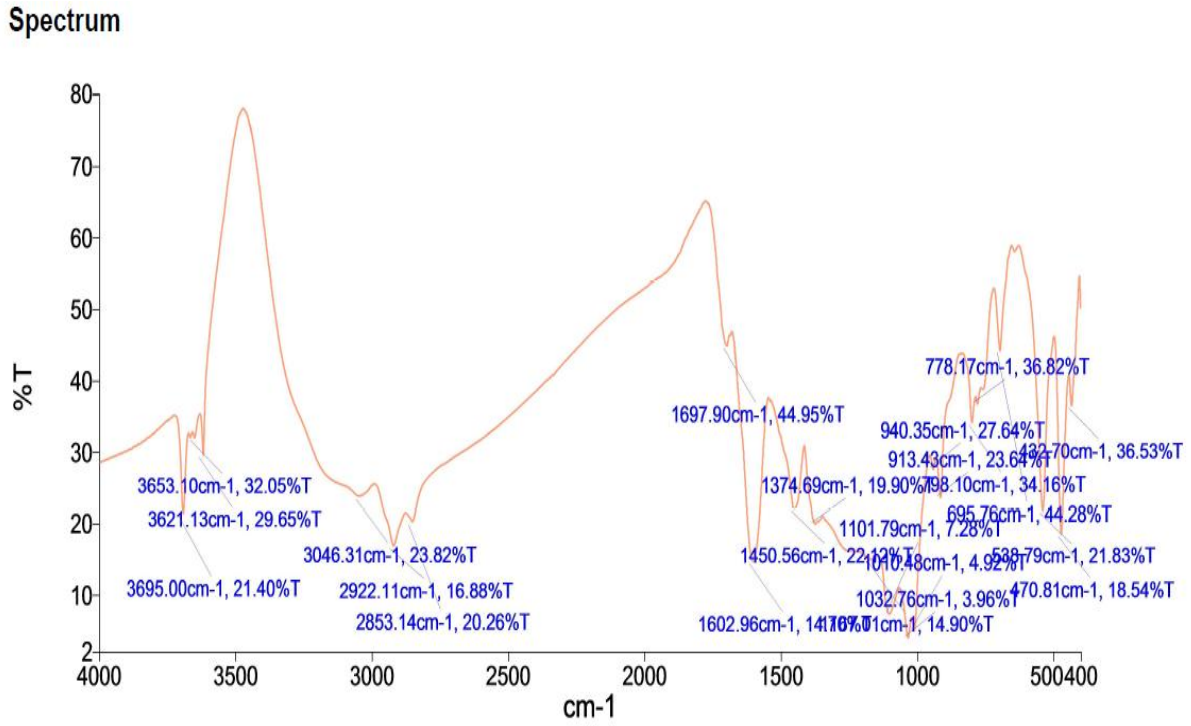

Figure A2-8: FTIR Transmittance spectra for SECL-1

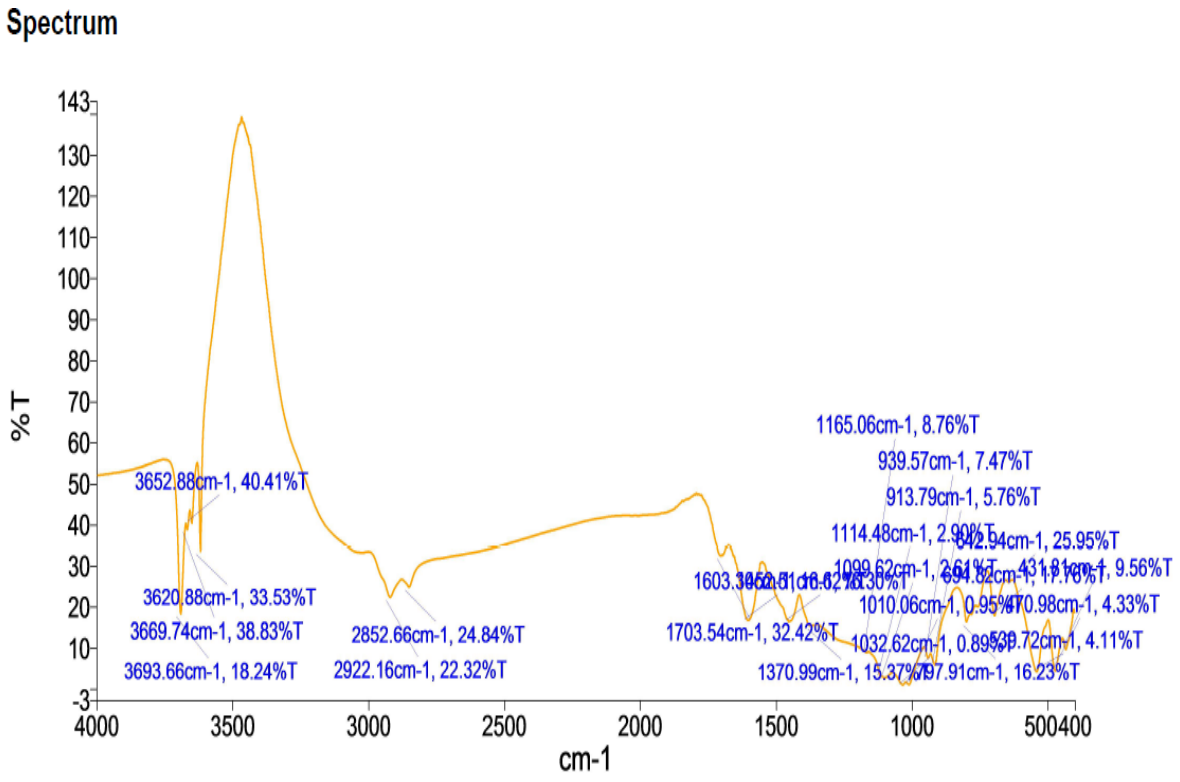

Figure A2-9:FTIR Transmittance spectra for SECL-2

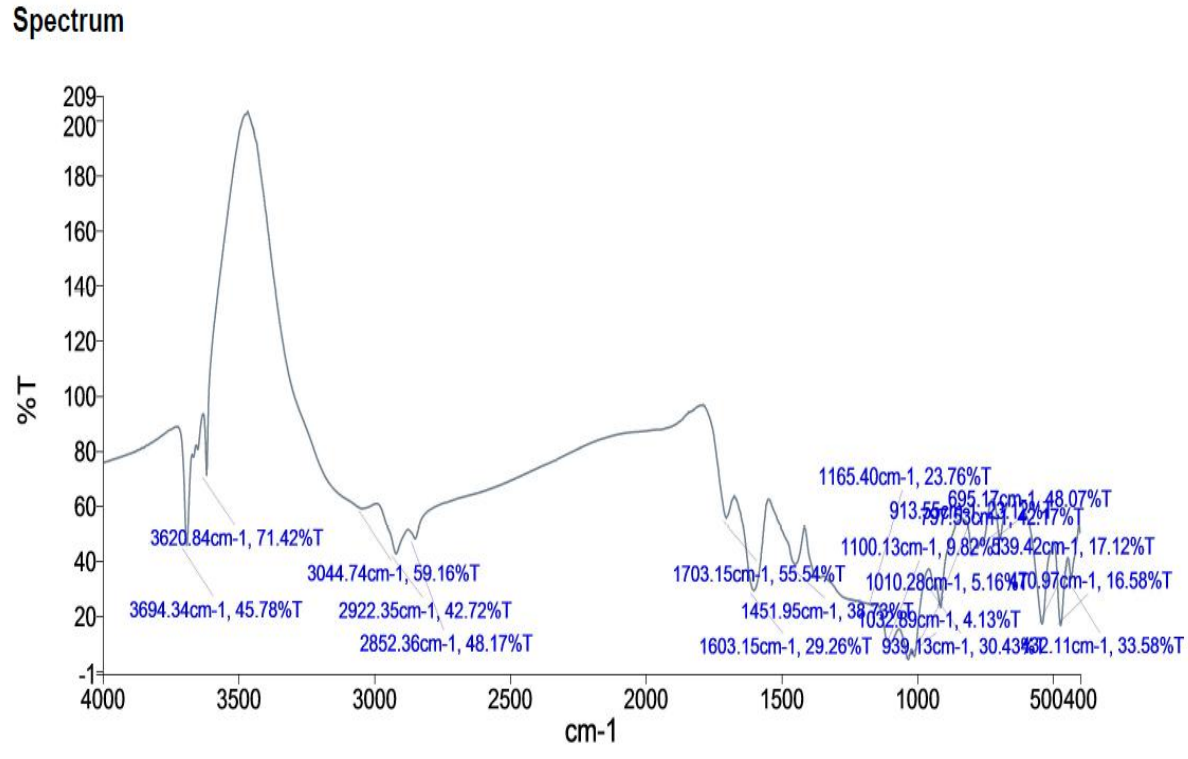

Figure A2-10: FTIR Transmittance spectra for SECL-3

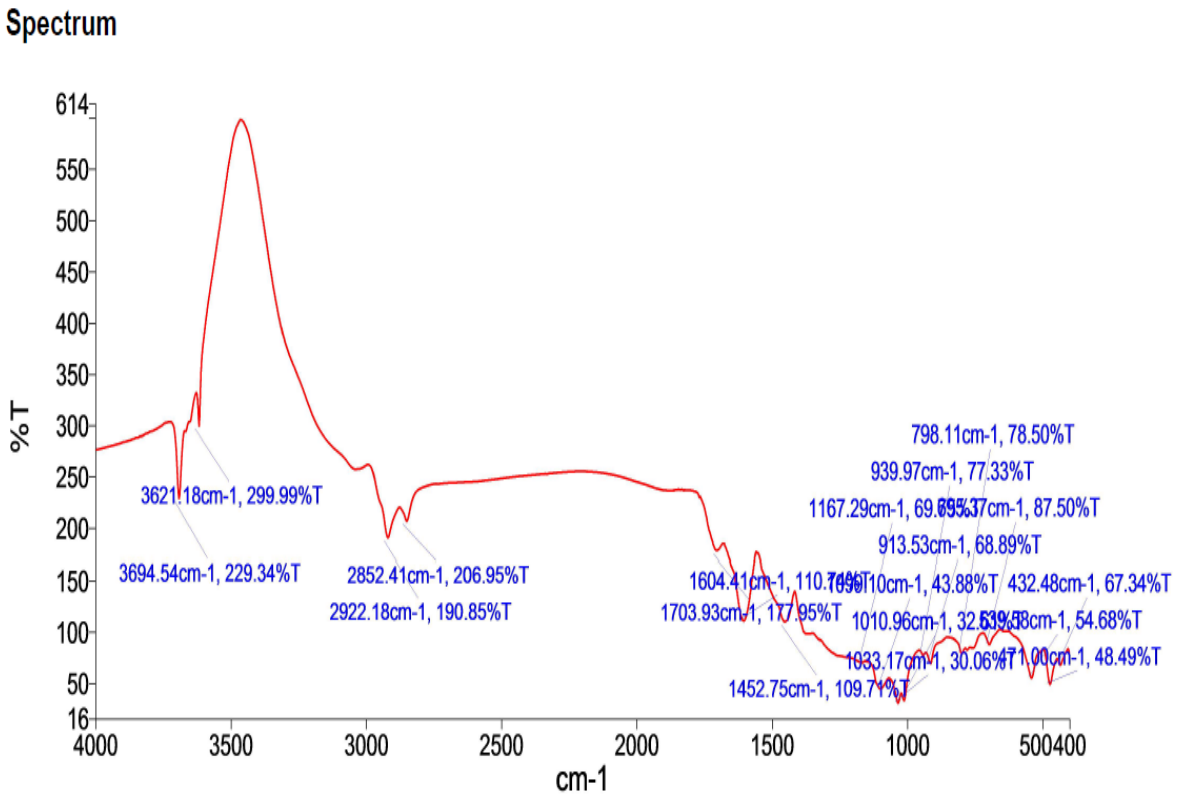

Figure A2-11: FTIR Transmittance spectra for SECL-4

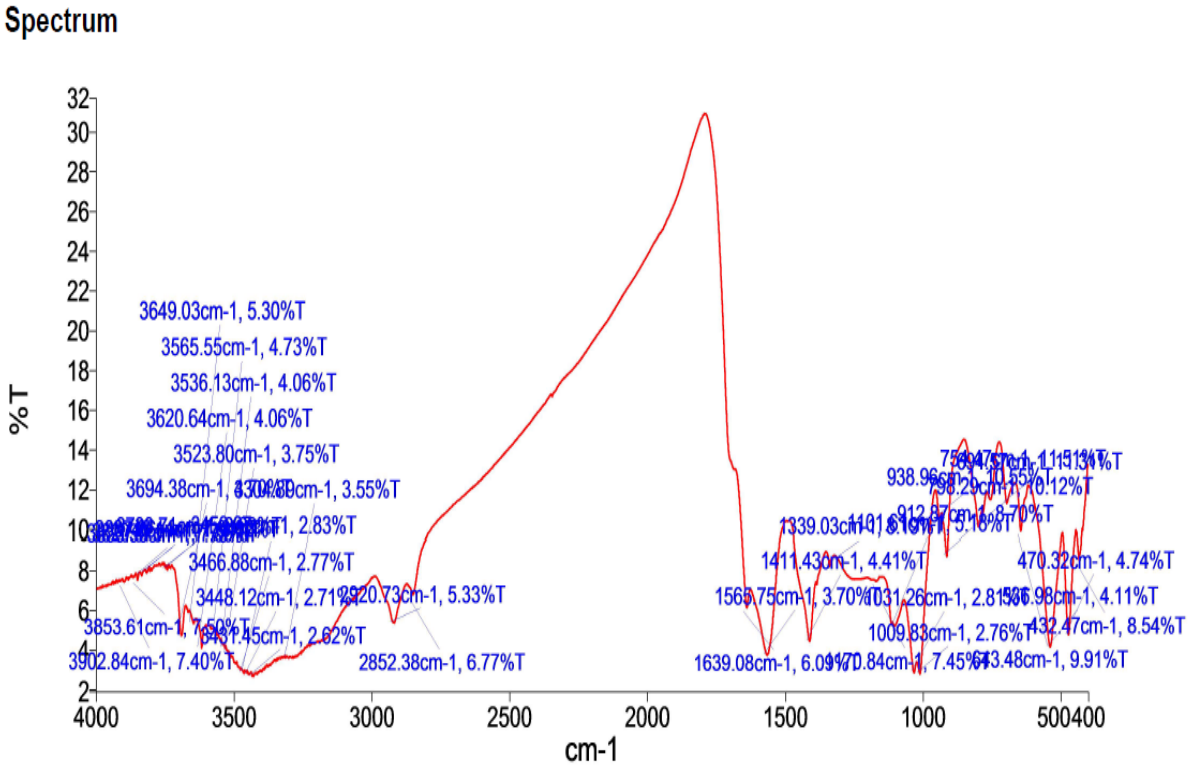

Figure A2-12: FTIR Transmittance spectra for SCCL – 5

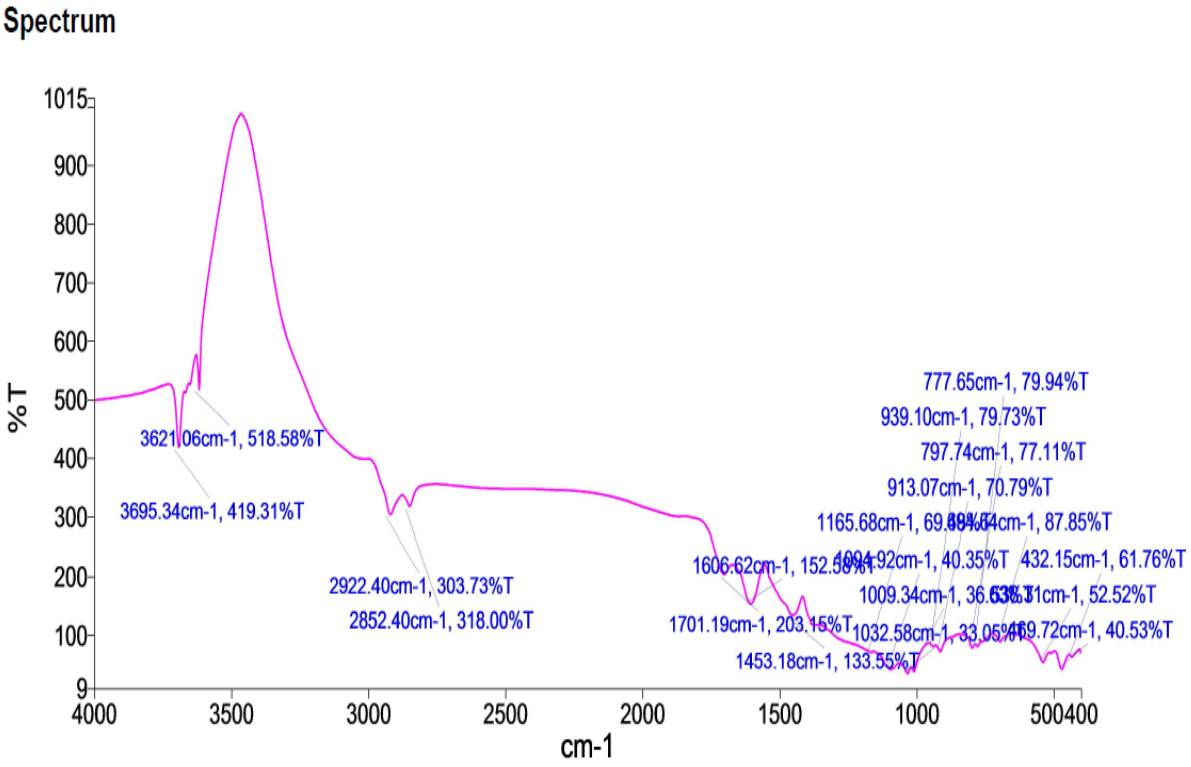

Figure A2-13: FTIR Transmittance spectra for SECL-6

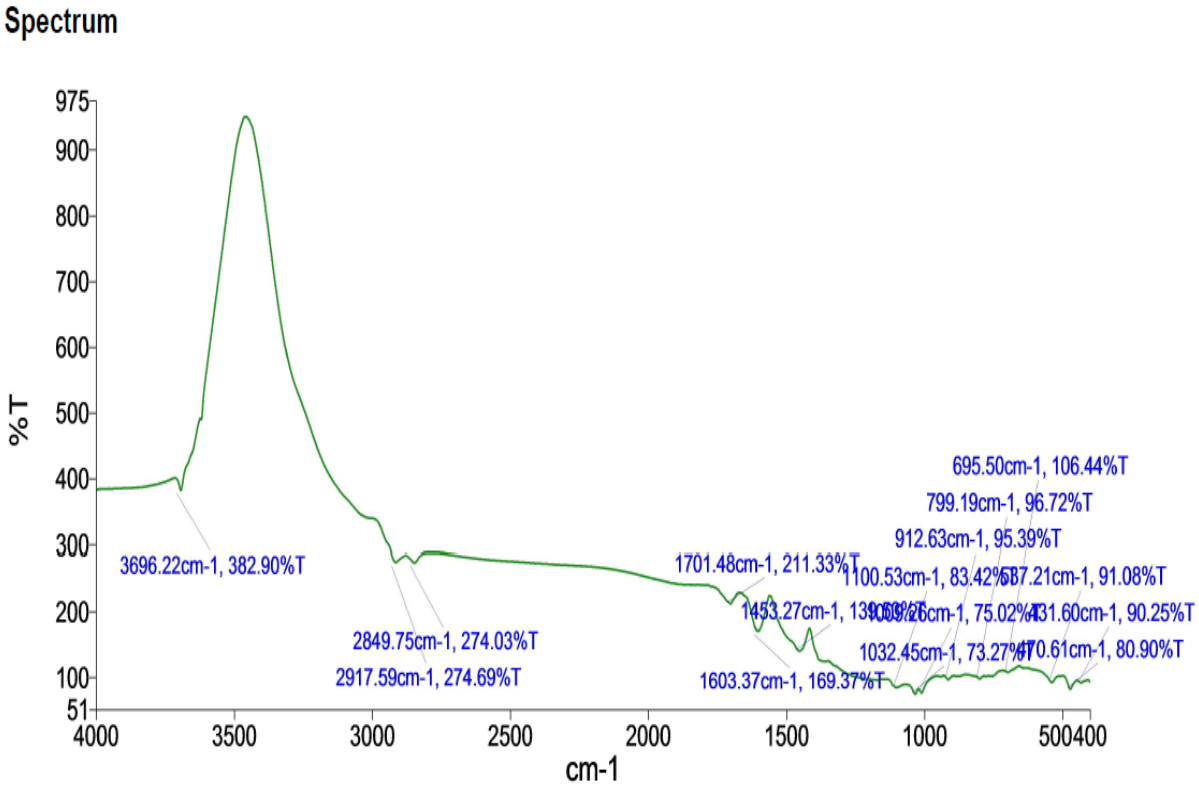

Figure A2-14: FTIR Transmittance spectra for SECL-7

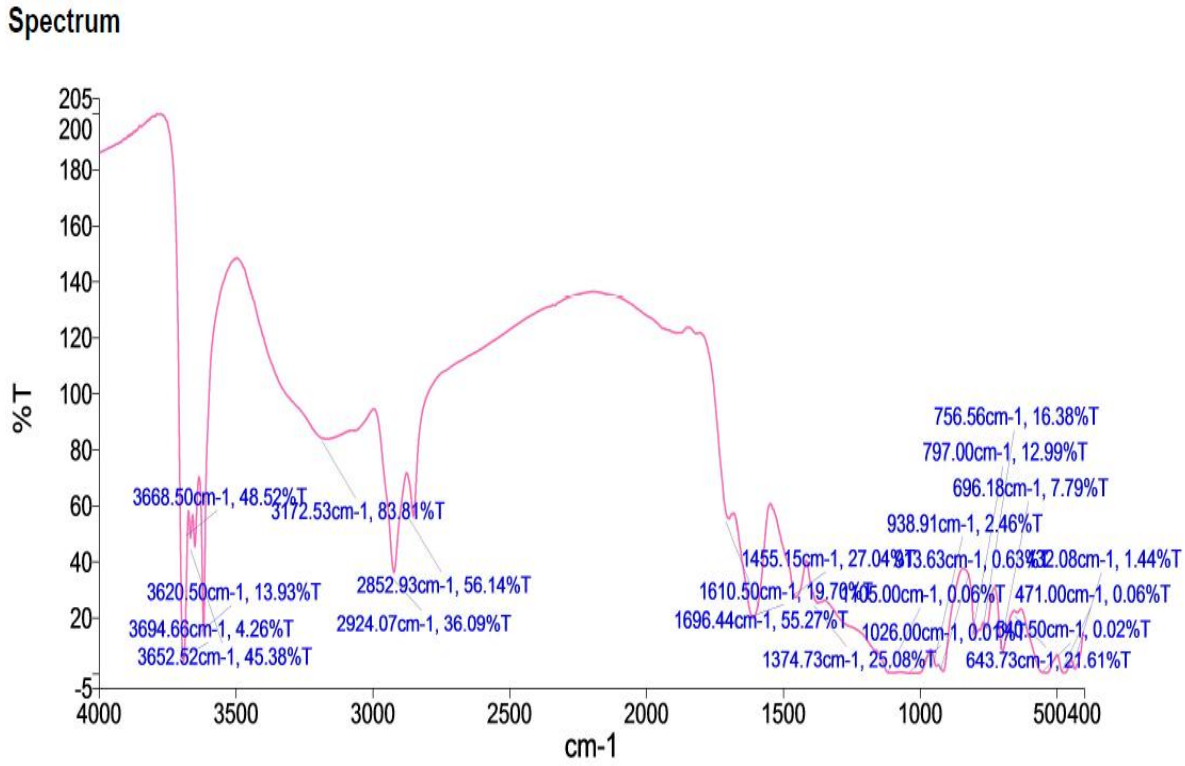

Figure A2-15: FTIR Transmittance spectra for CCL-1

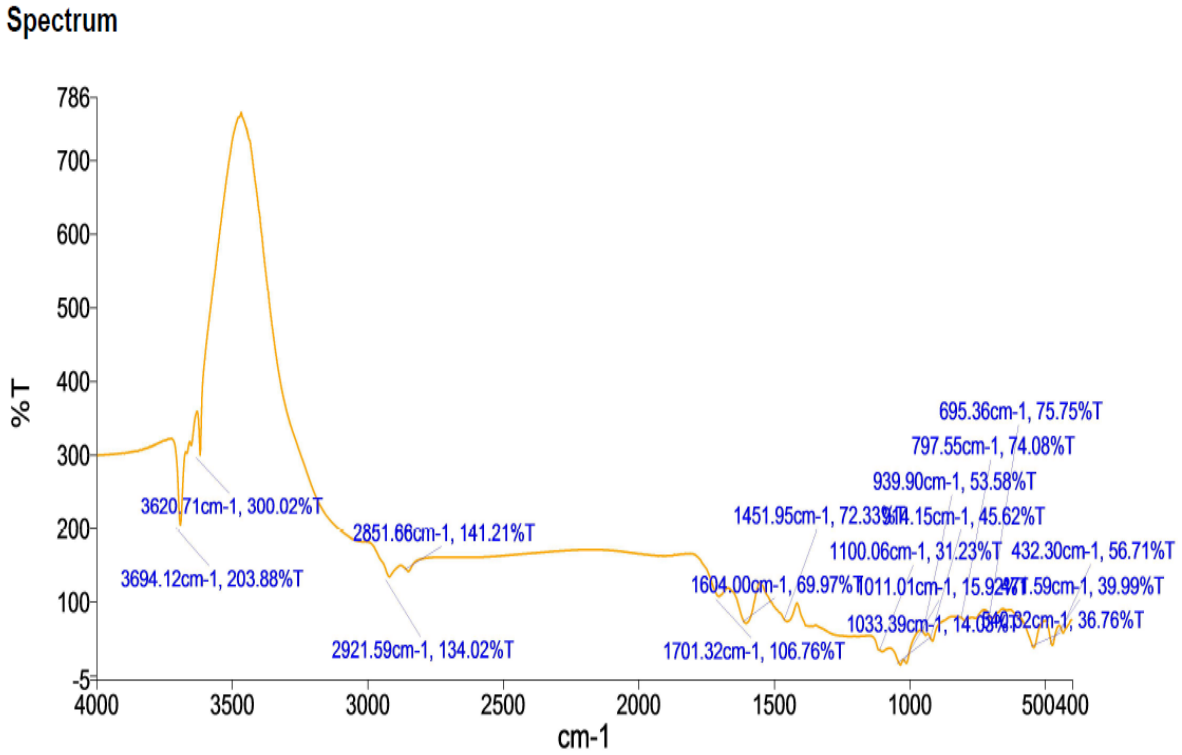

Figure A2-16: FTIR Transmittance spectra for CCL-2

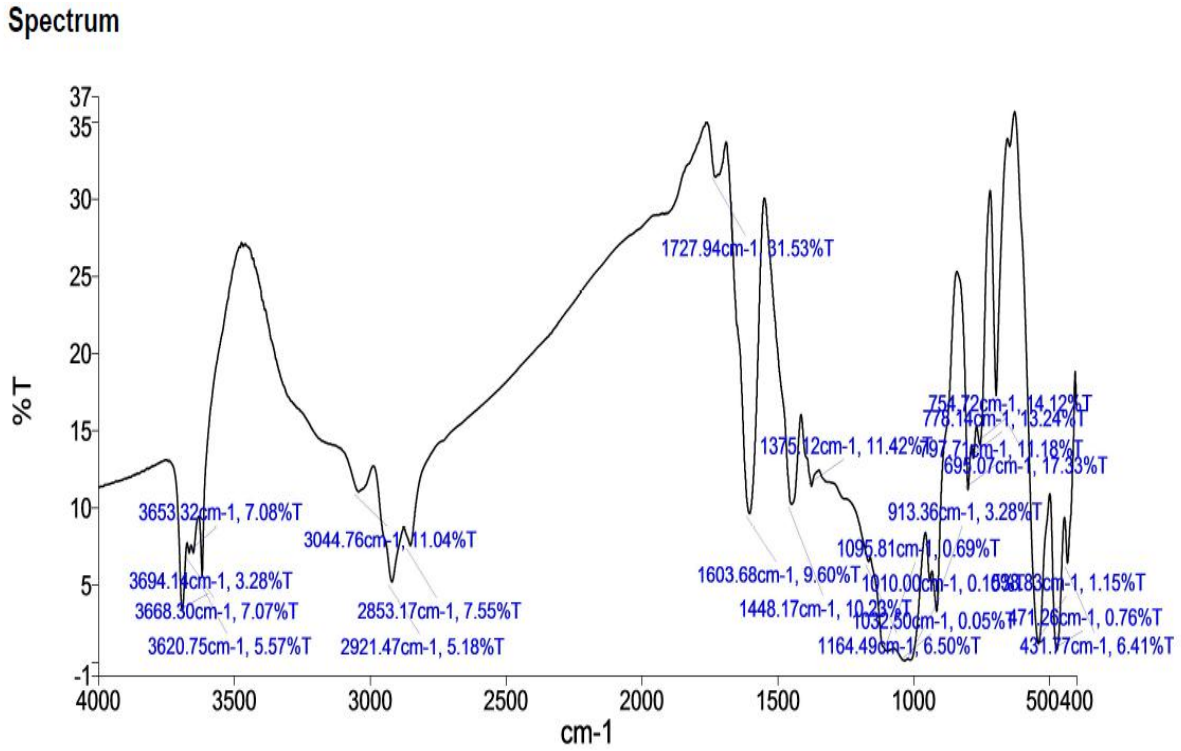

Figure A2-17: FTIR Transmittance spectra for ECL-1

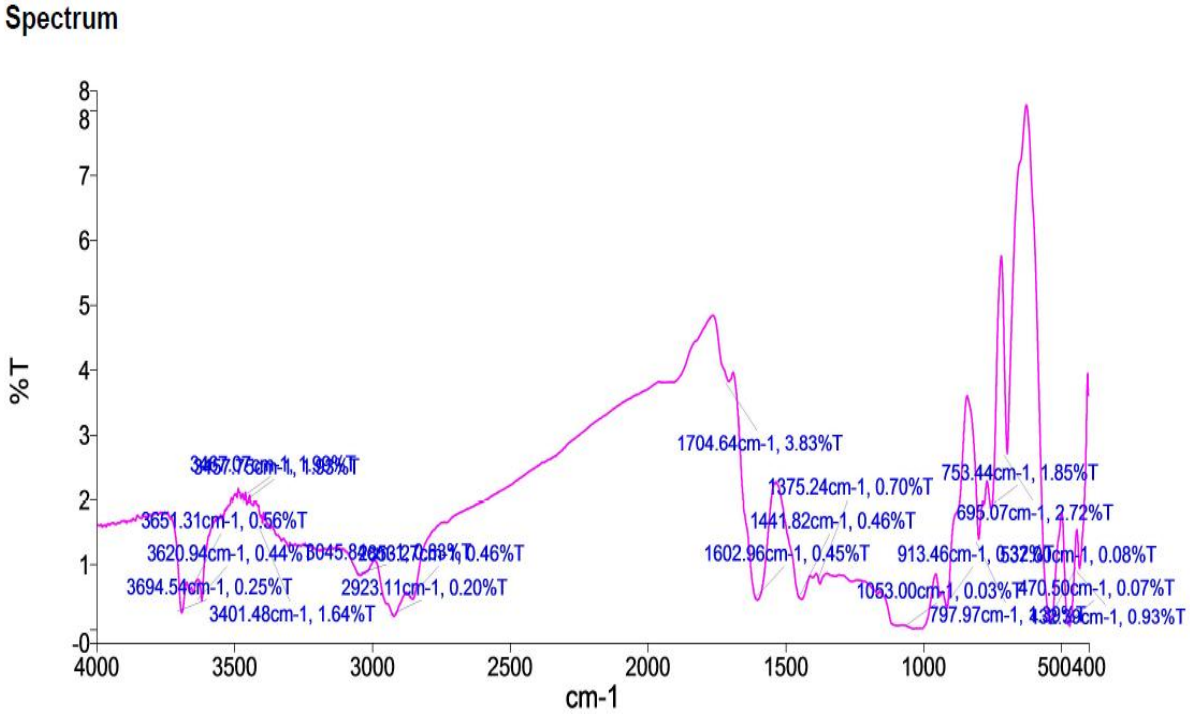

Figure A2-18: FTIR Transmittance spectra for ECL-2

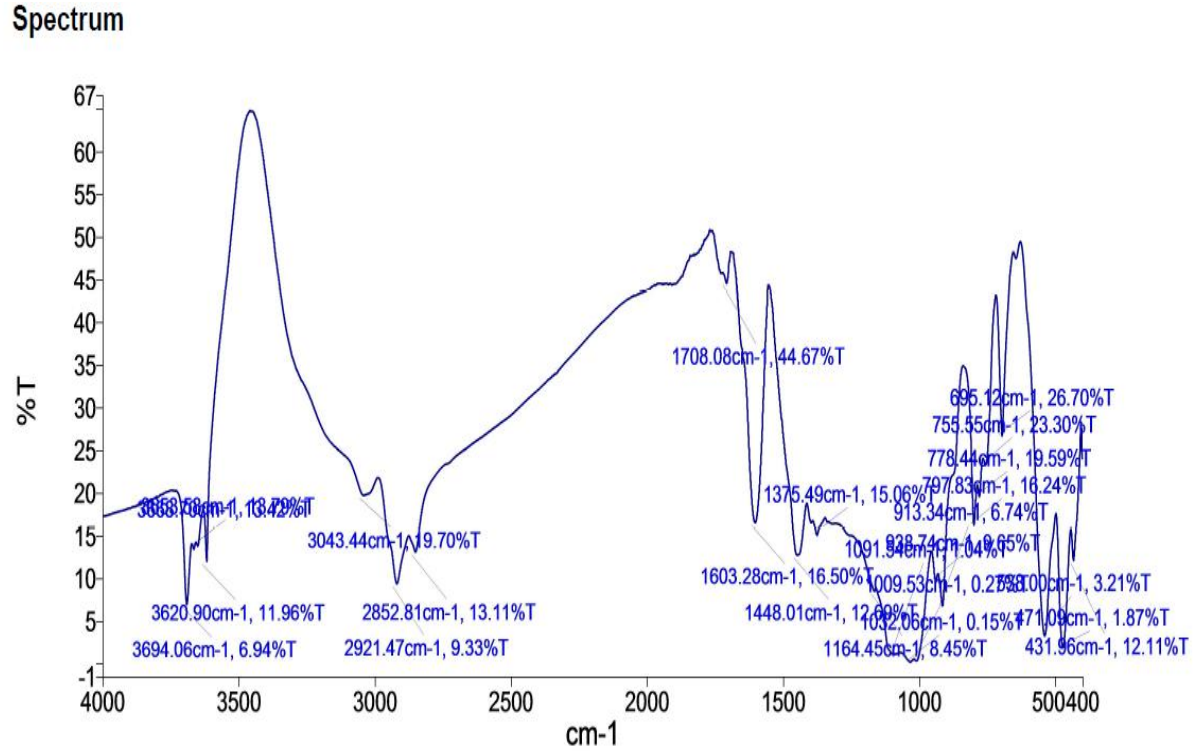

Figure A2-19: FTIR Transmittance spectra for ECL – 3

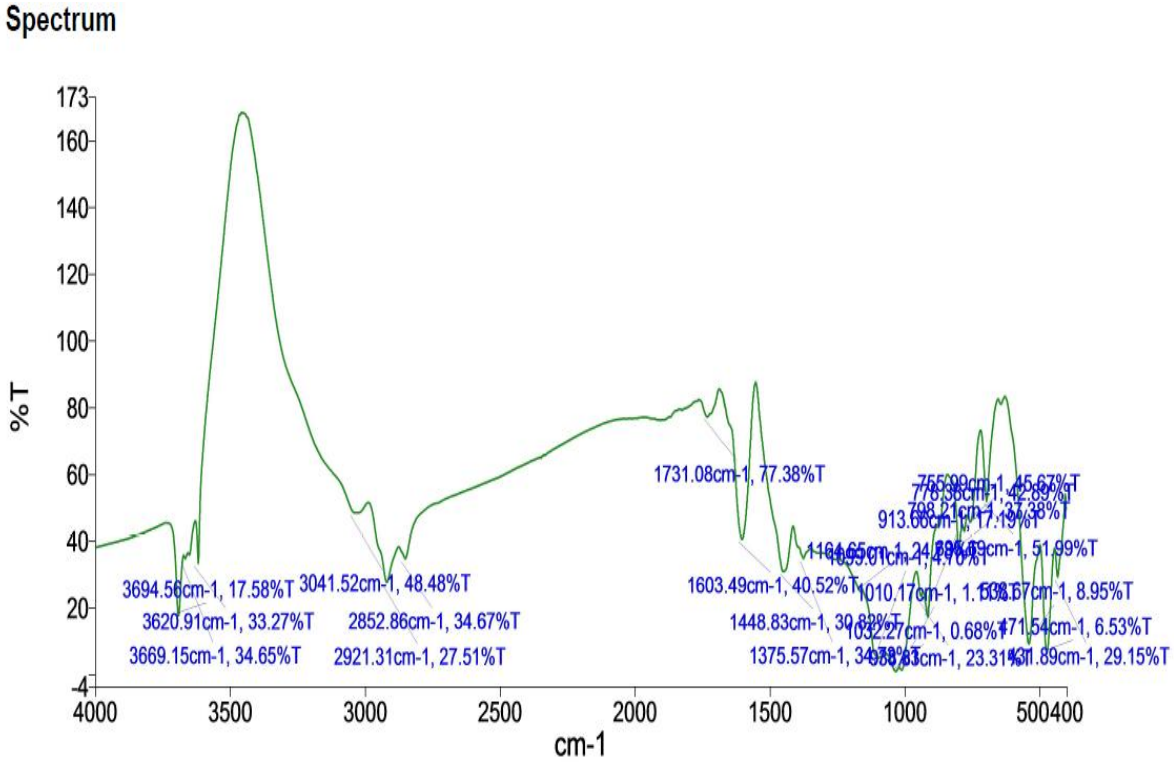

Figure A2-20: FTIR Transmittance spectra for ECL – 4

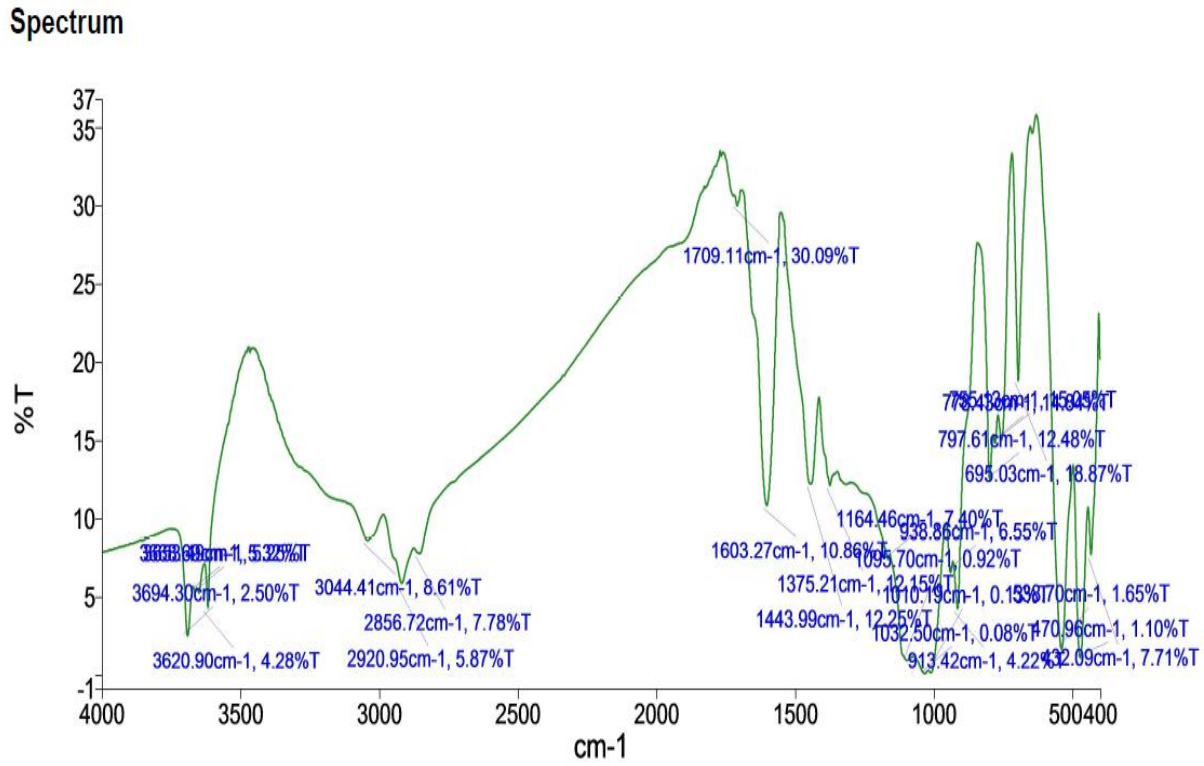

Figure A2-21: FTIR Transmittance spectra for ECL-5

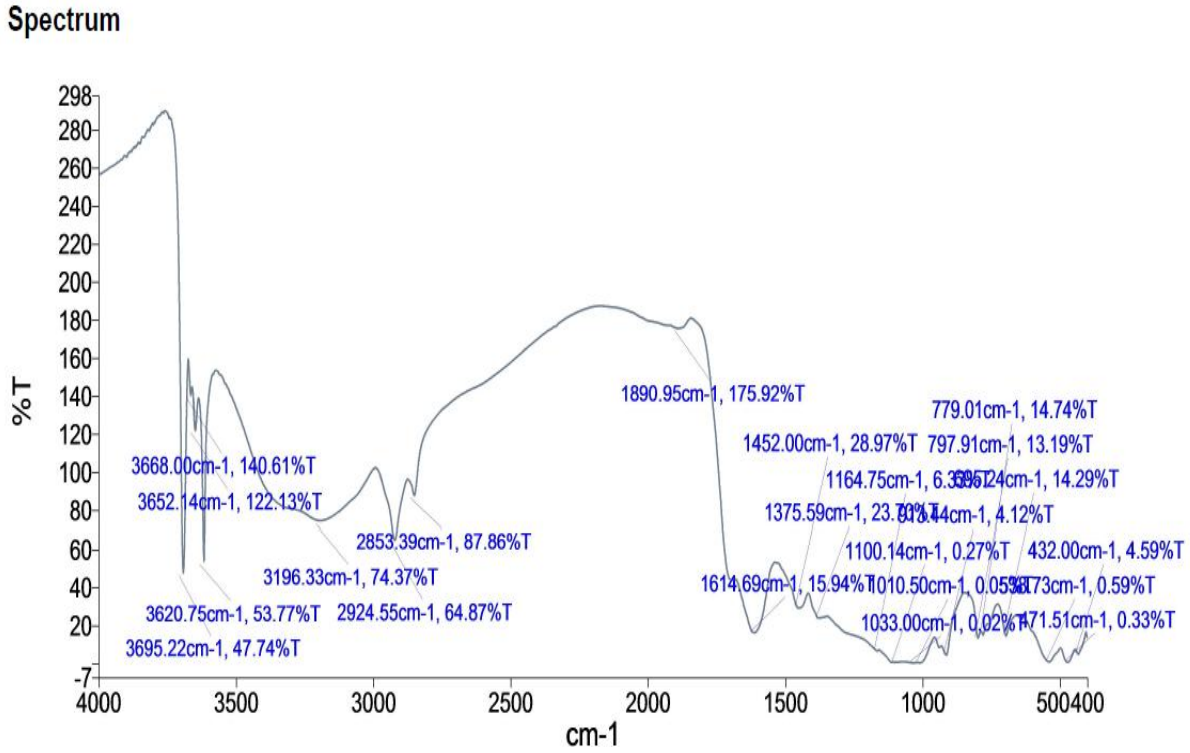

Figure A2-22: FTIR Transmittance spectra for ECL-6

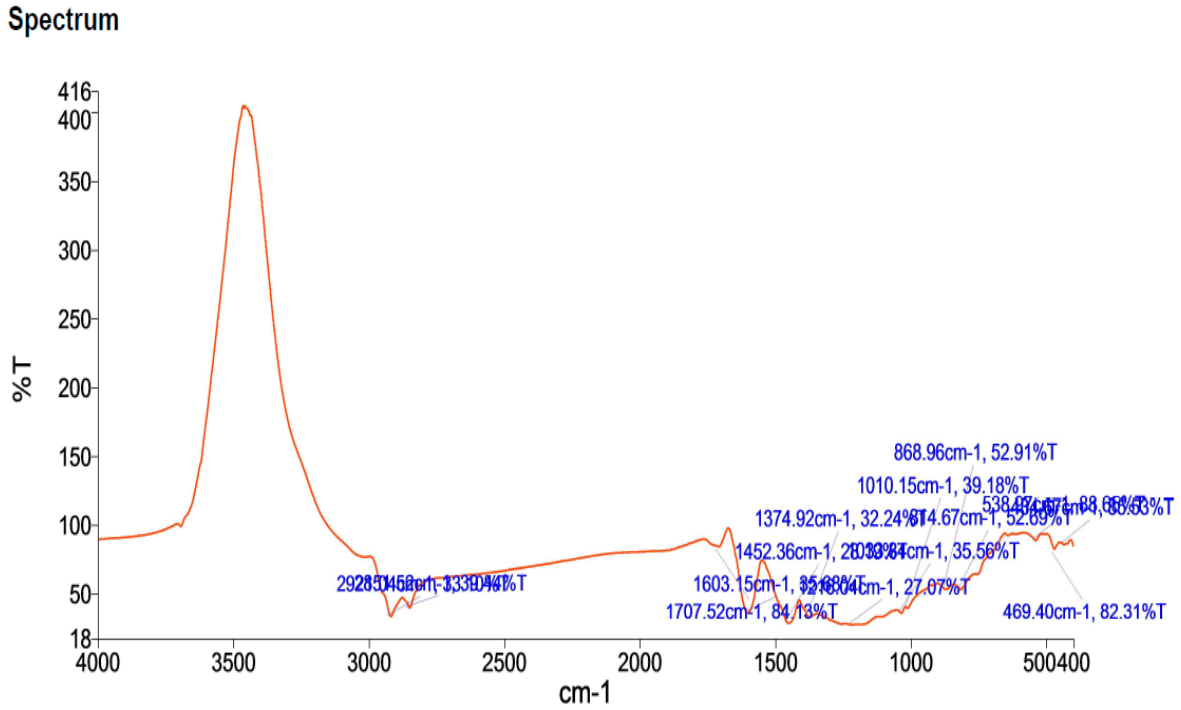

Figure A2-23: FTIR Transmittance spectra for NEC-1

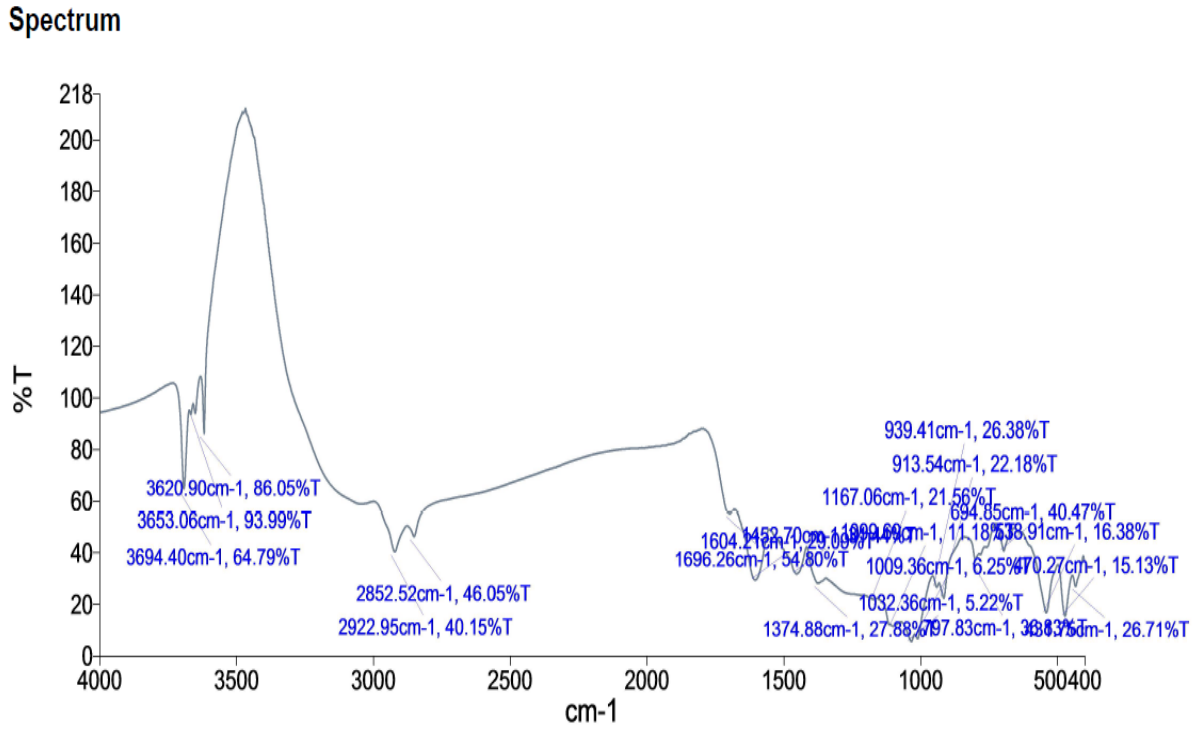

Figure A2-24: FTIR Transmittance spectra for MCL – 1

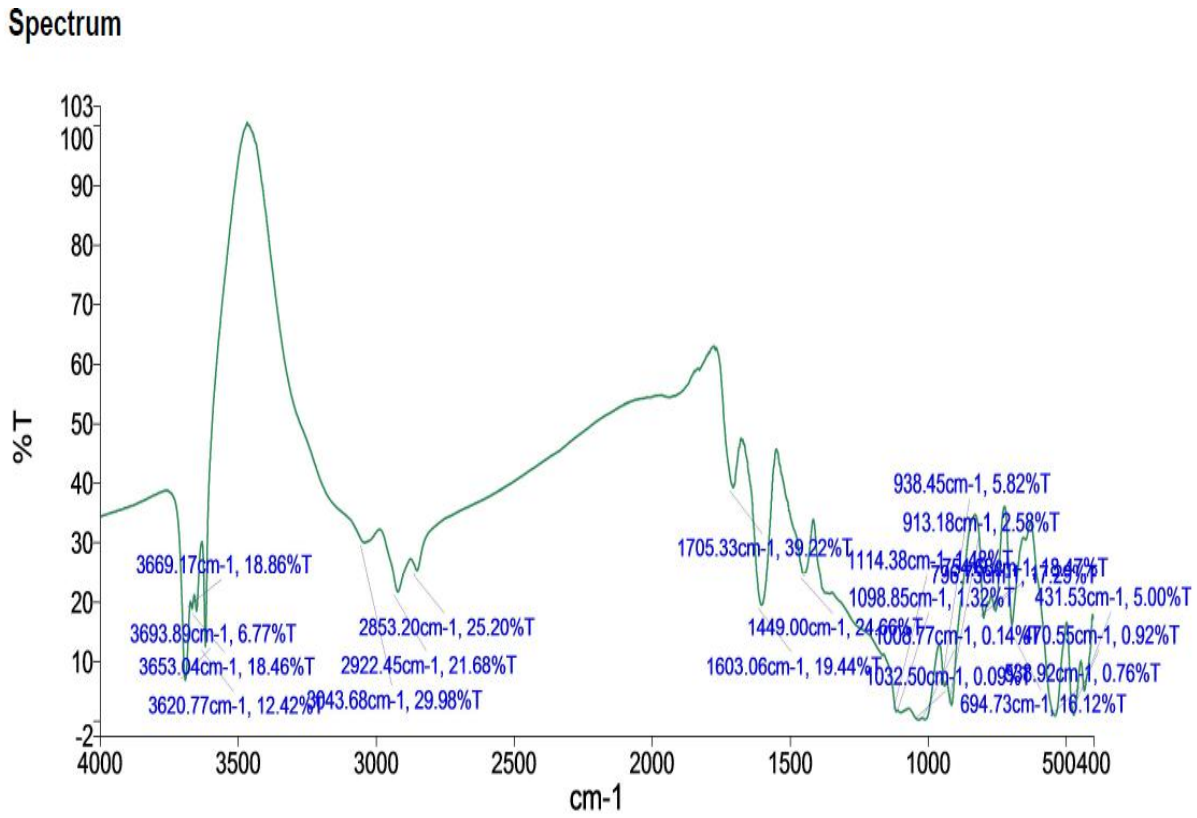

Figure A2-25: FTIR Transmittance spectra for MCL – 2

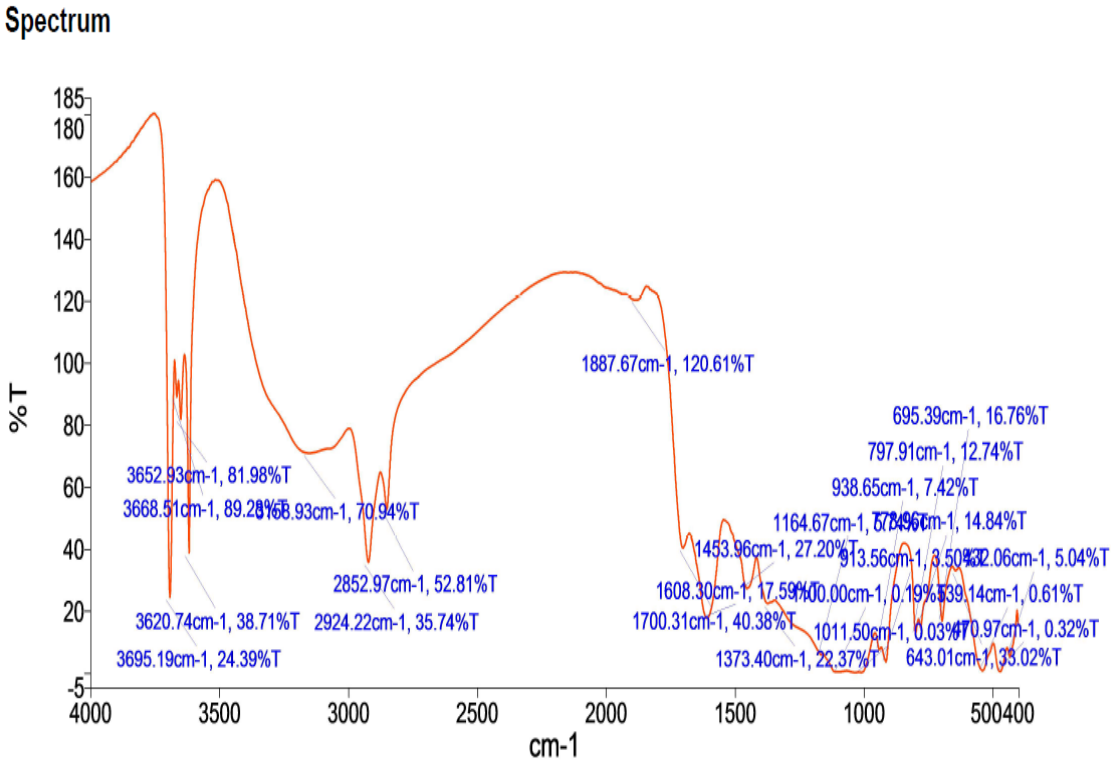

Figure A2-26: FTIR Transmittance spectra for MCL-3

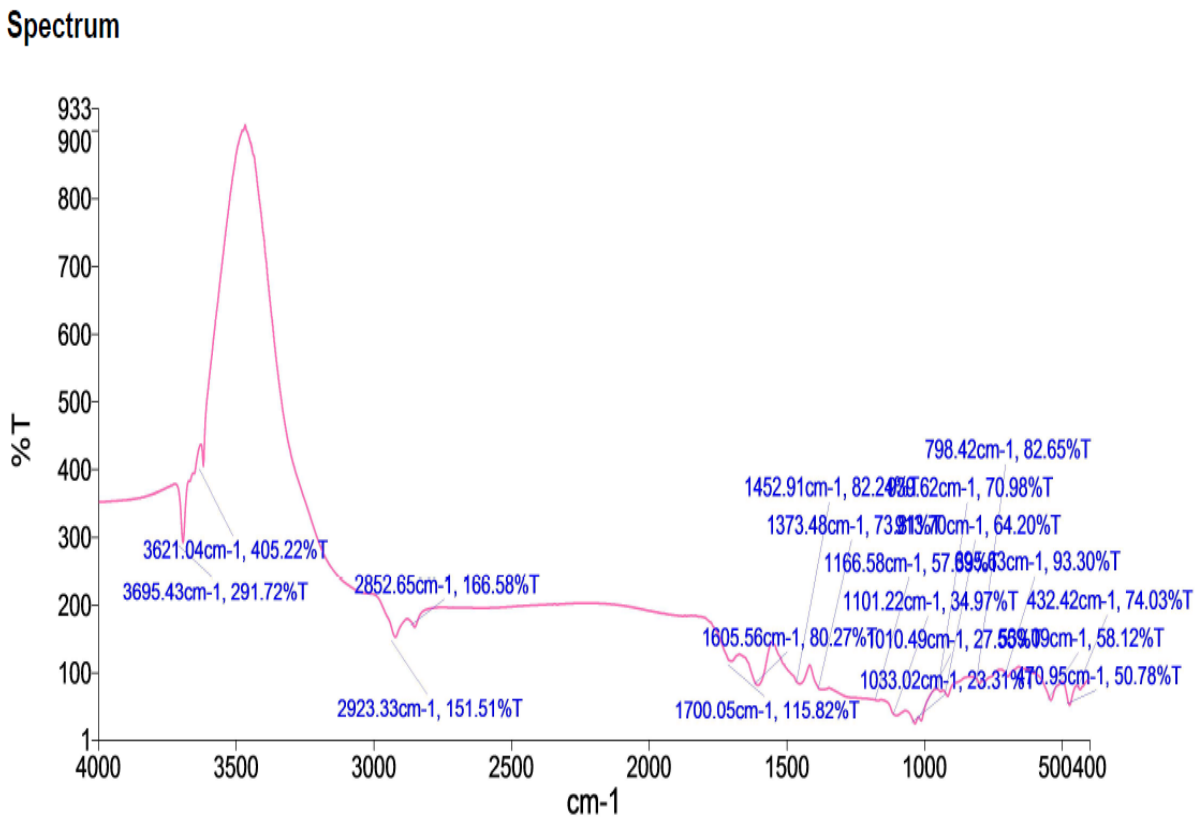

Figure A2-27: FTIR Transmittance spectra for MCL – 4

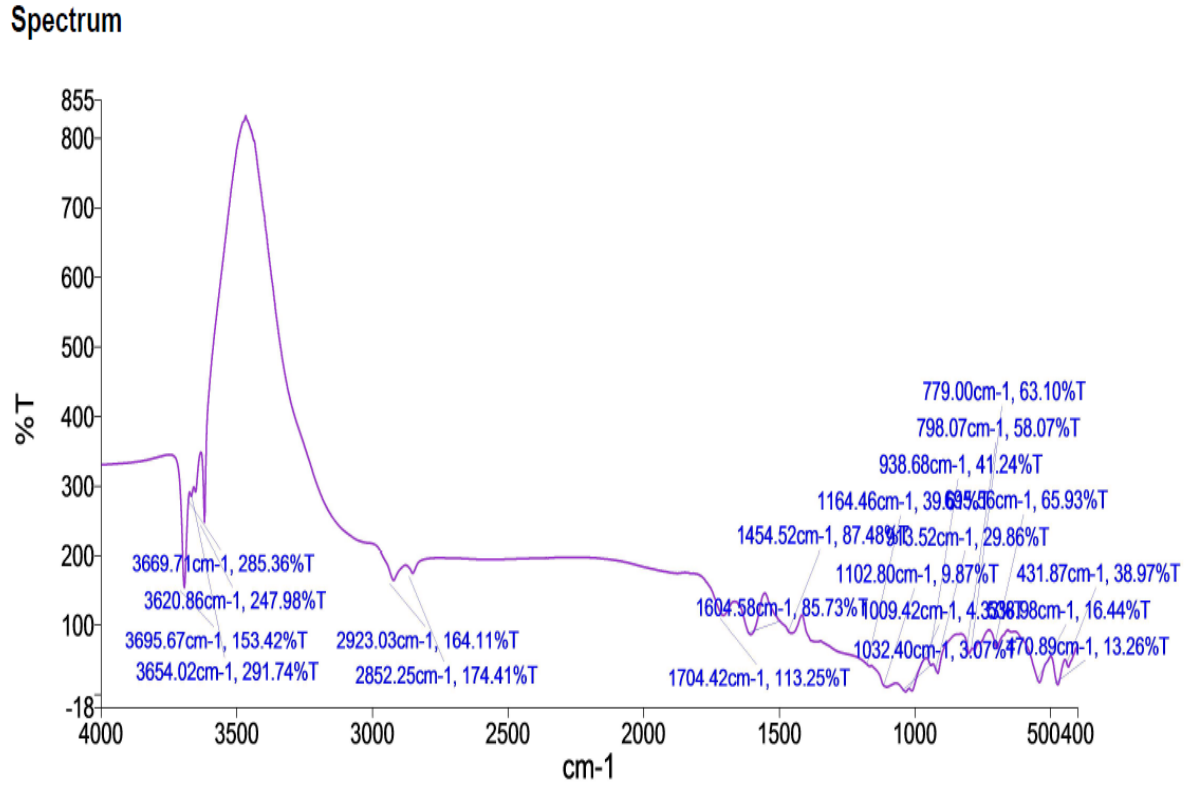

Figure A2-28: FTIR Transmittance spectra for MCL – 5

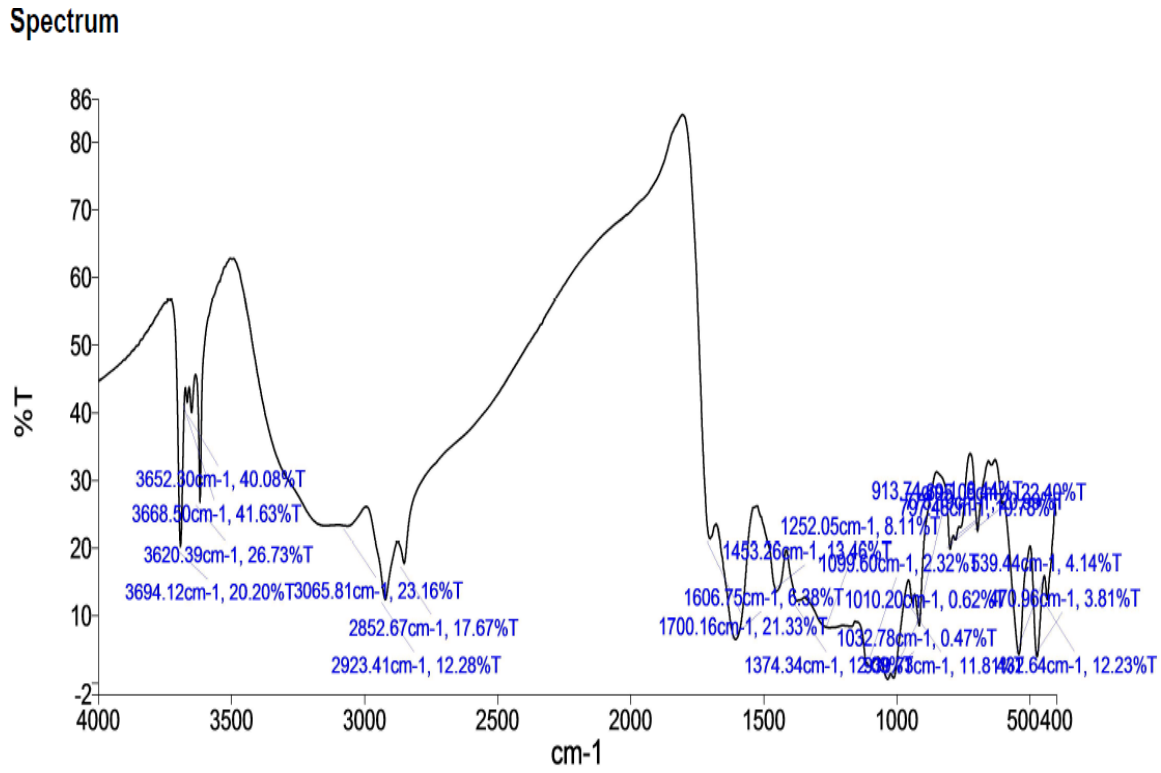

Figure A2-29: FTIR Transmittance spectra for MCL – 6

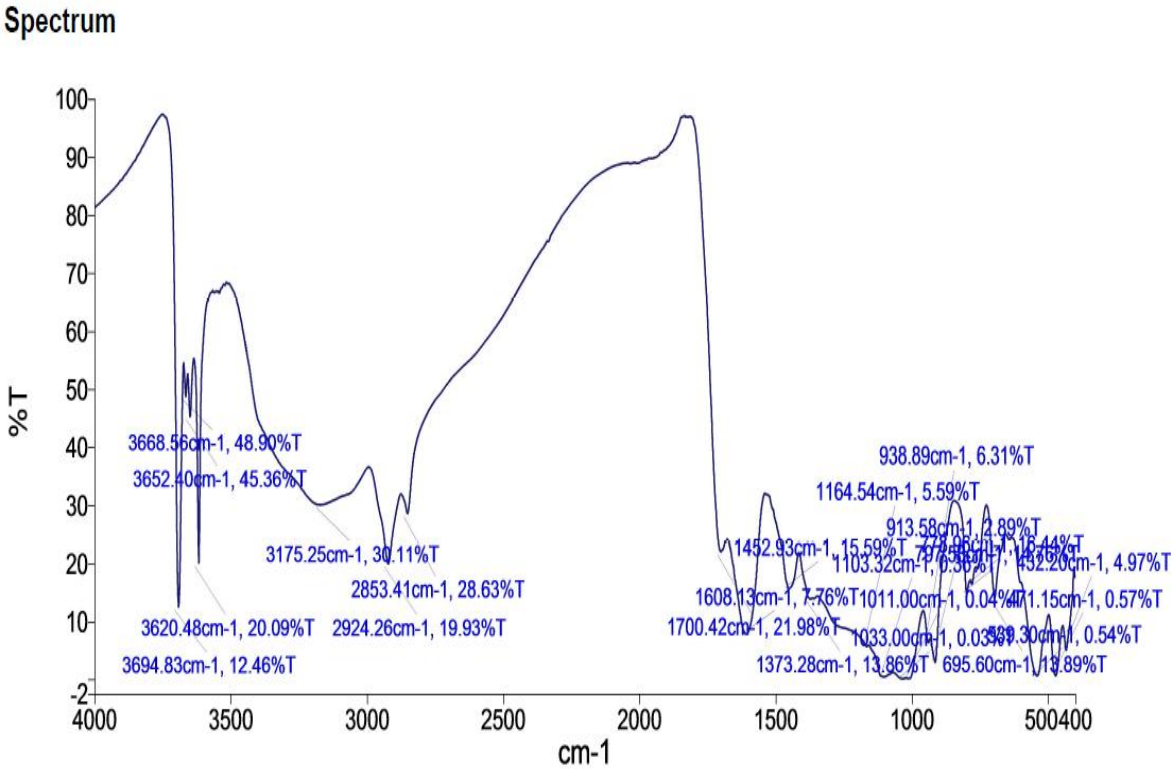

Figure A2-30: FTIR Transmittance spectra for MCL – 7

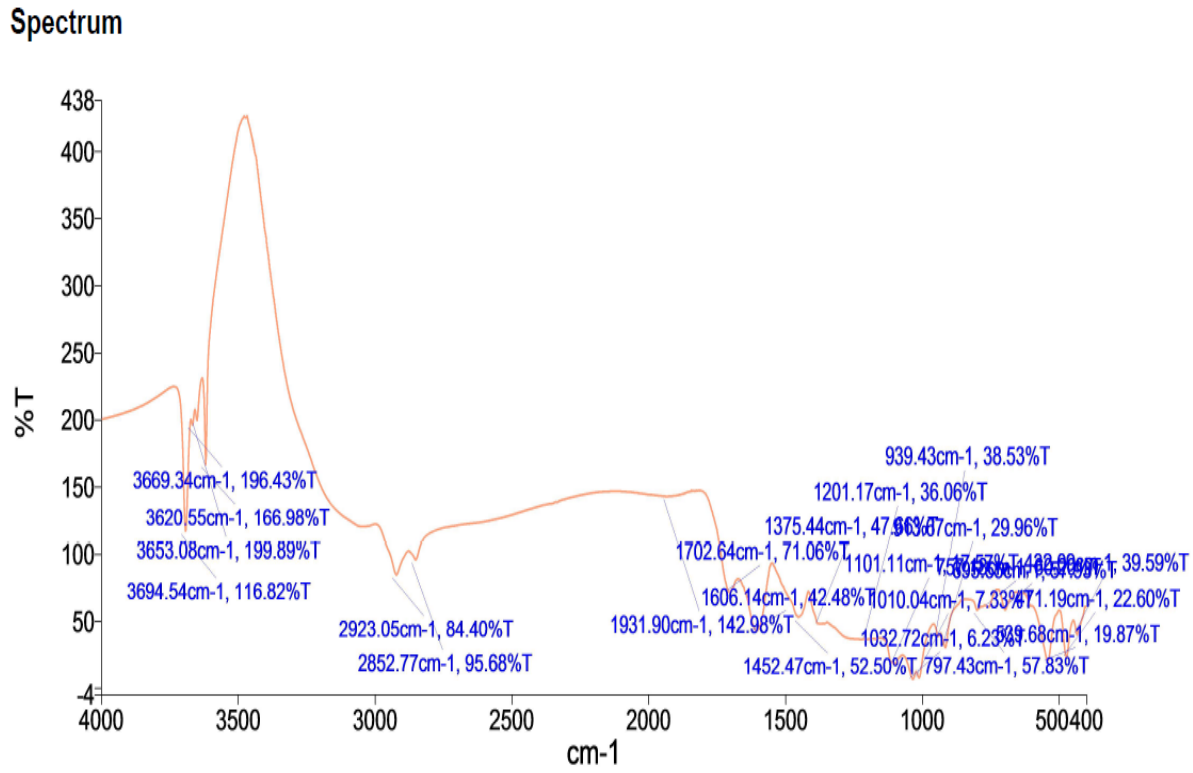

Figure A2-31: FTIR Transmittance spectra for MCL – 8

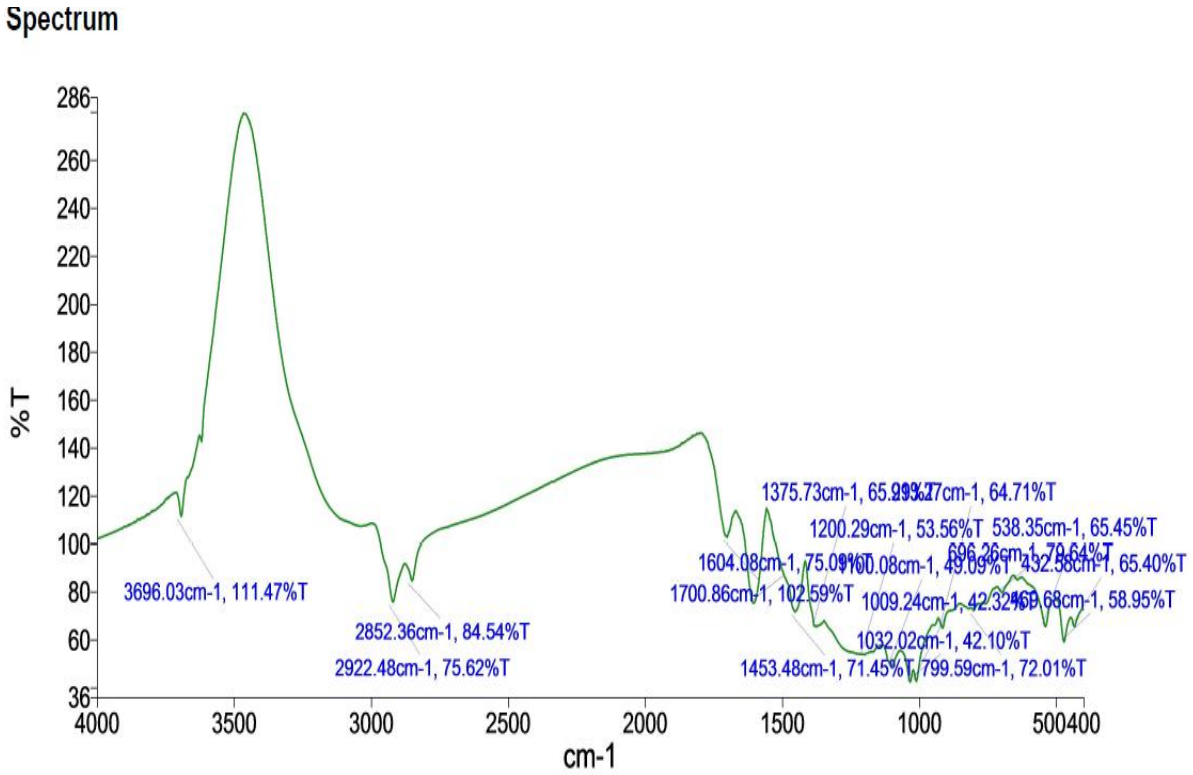

Figure A2-32: FTIR Transmittance spectra for MCL – 9

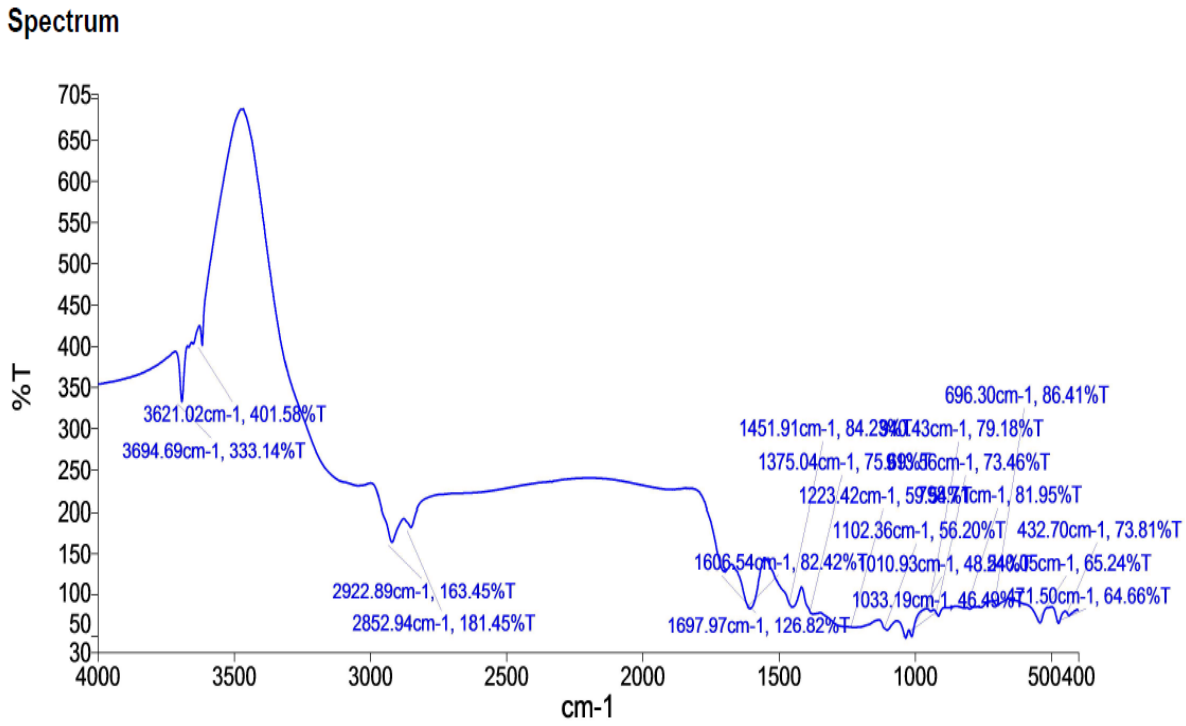

Figure A2-33: FTIR Transmittance spectra for MCL – 10

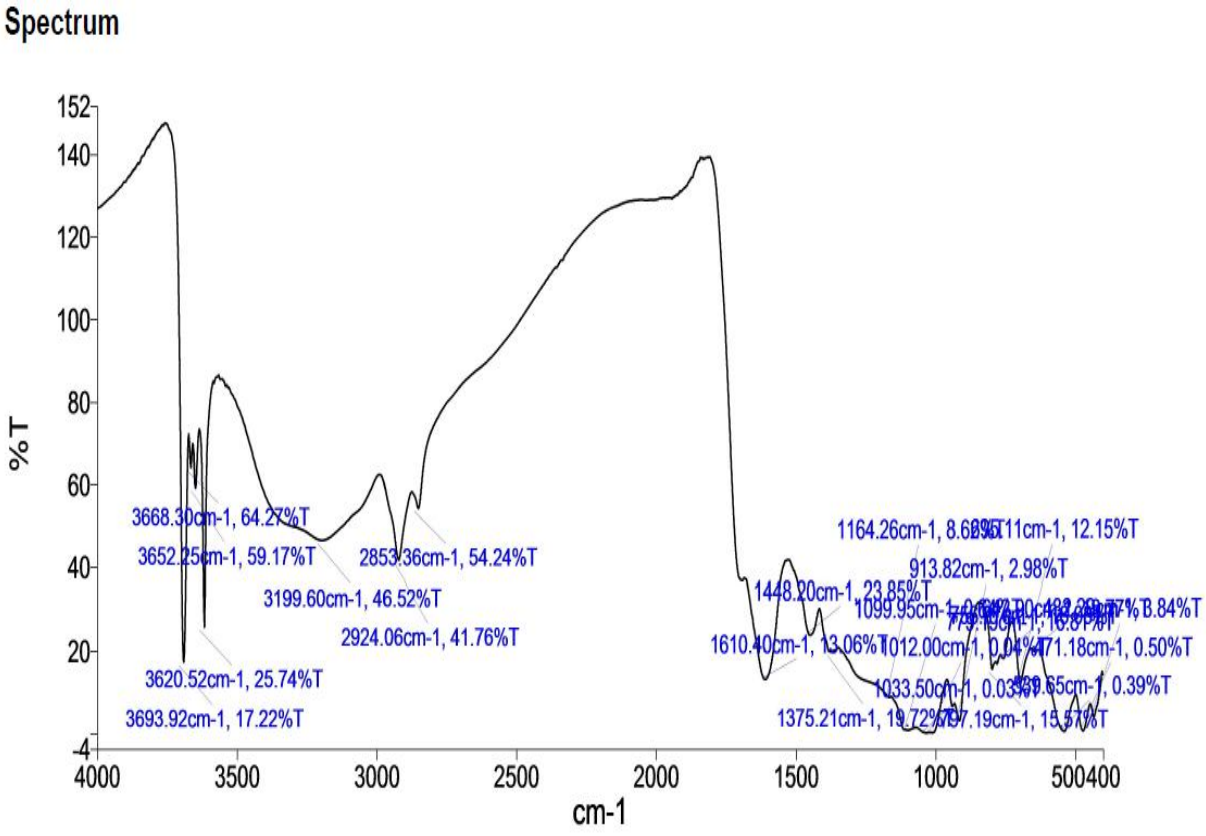

Figure A2-34: FTIR Transmittance spectra for MCL – 11

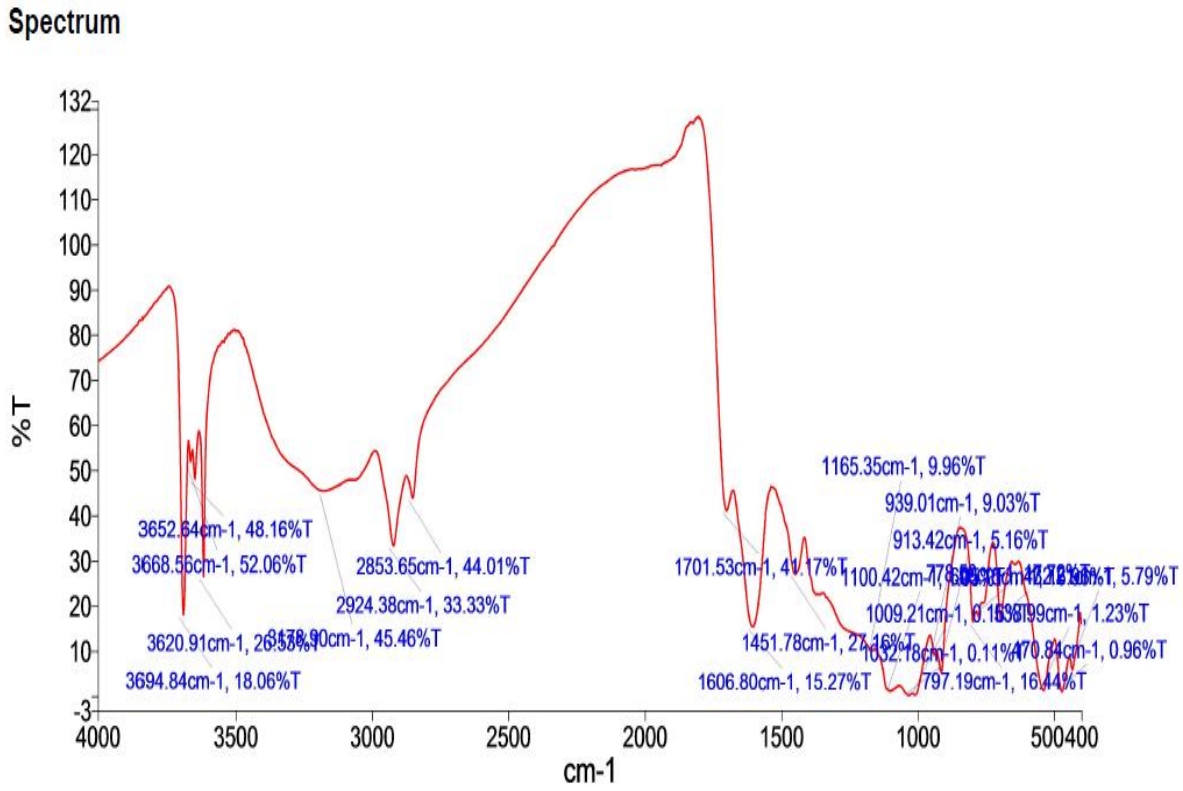

Figure A2-35: FTIR Transmittance spectra for MCL – 12

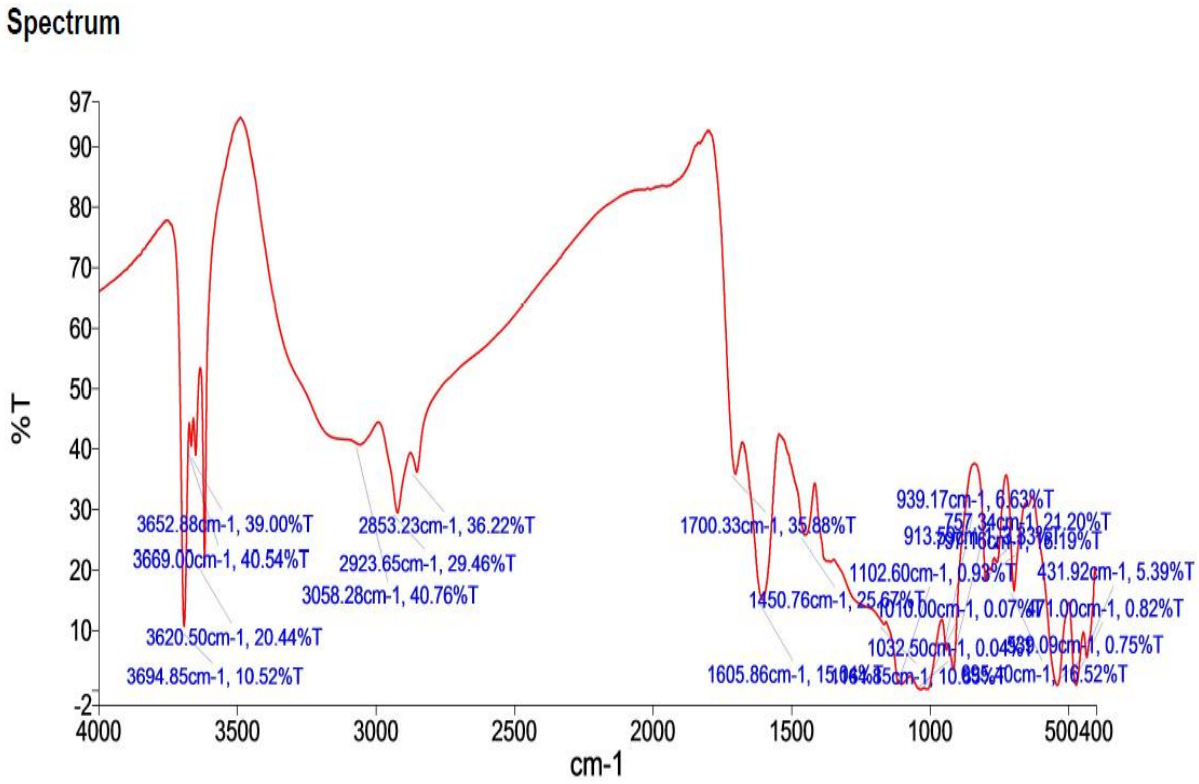

Figure A2-36: FTIR Transmittance spectra for MCL – 14

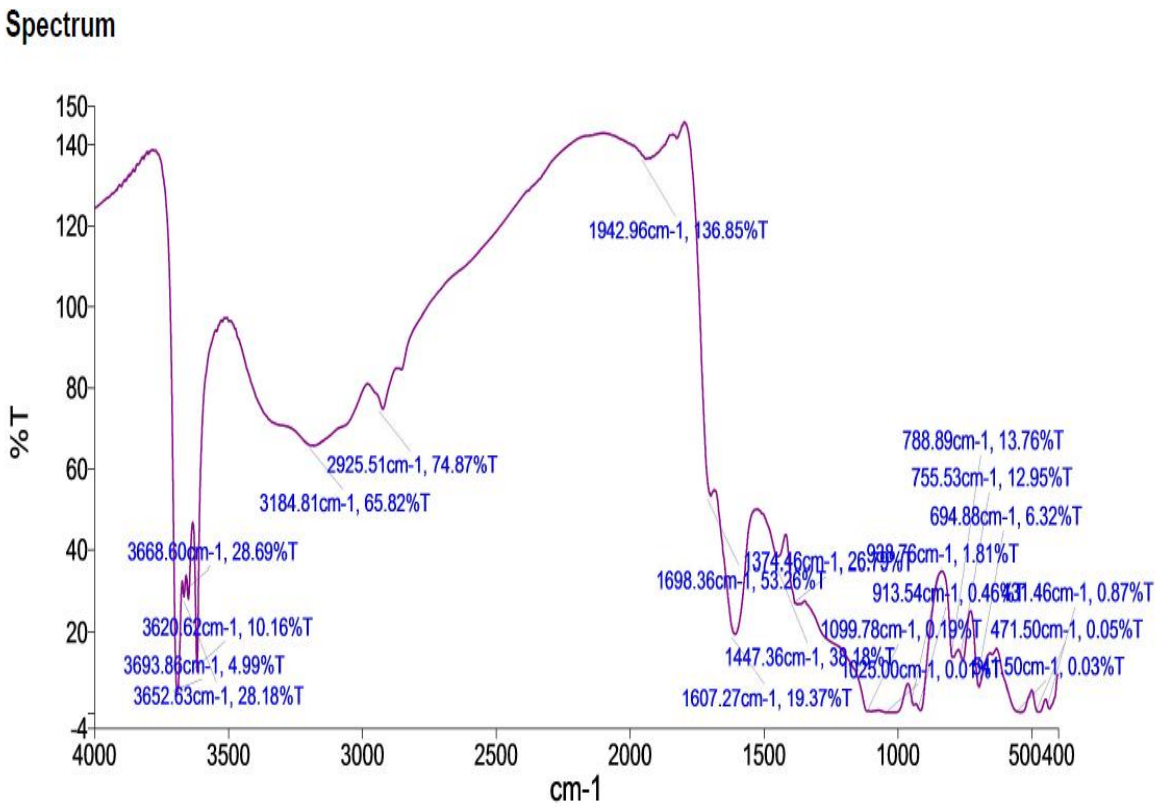

Figure A2-37: FTIR Transmittance spectra for MCL – 15

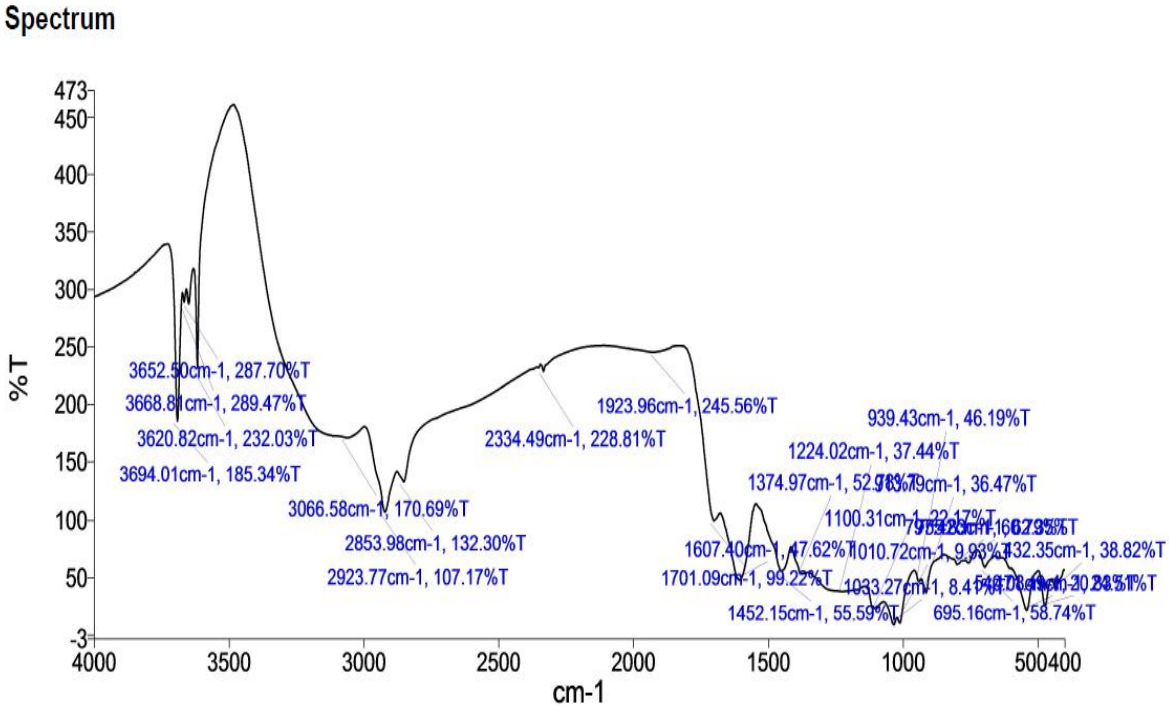

Figure A2-38: FTIR Transmittance spectra for MCL – 16

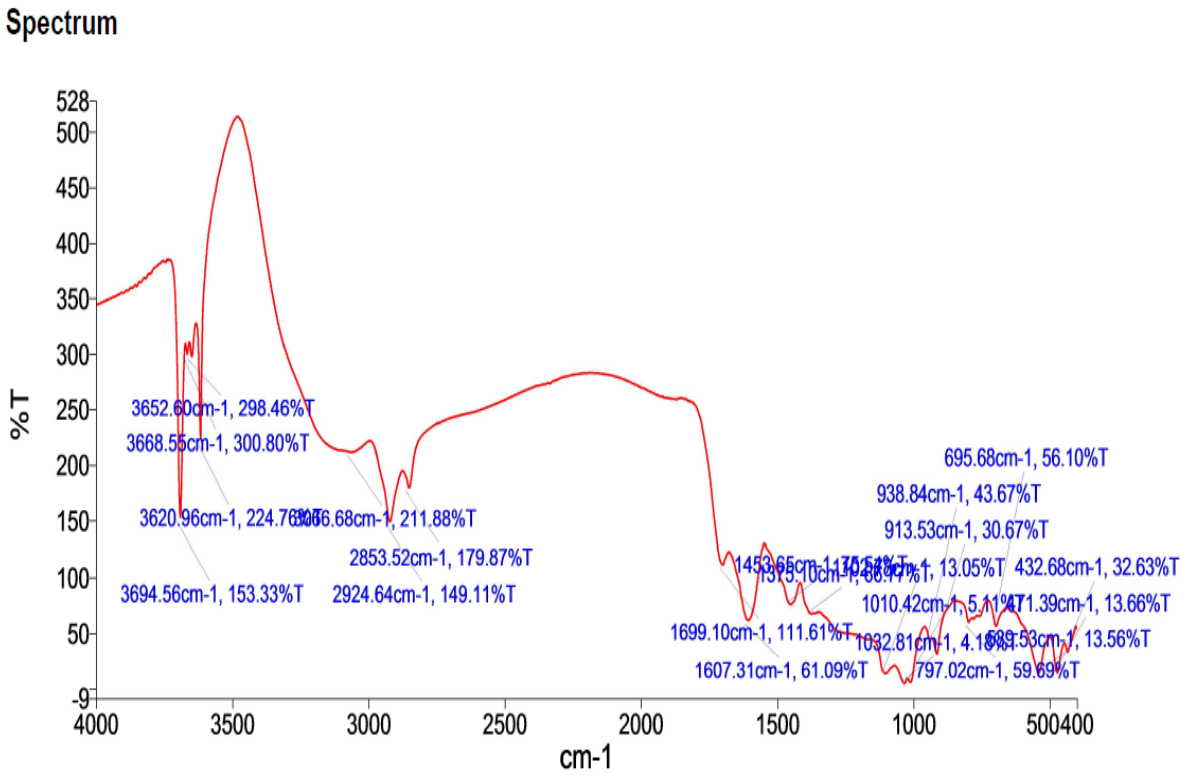

Figure A2-40: FTIR Transmittance spectra for MCL – 17

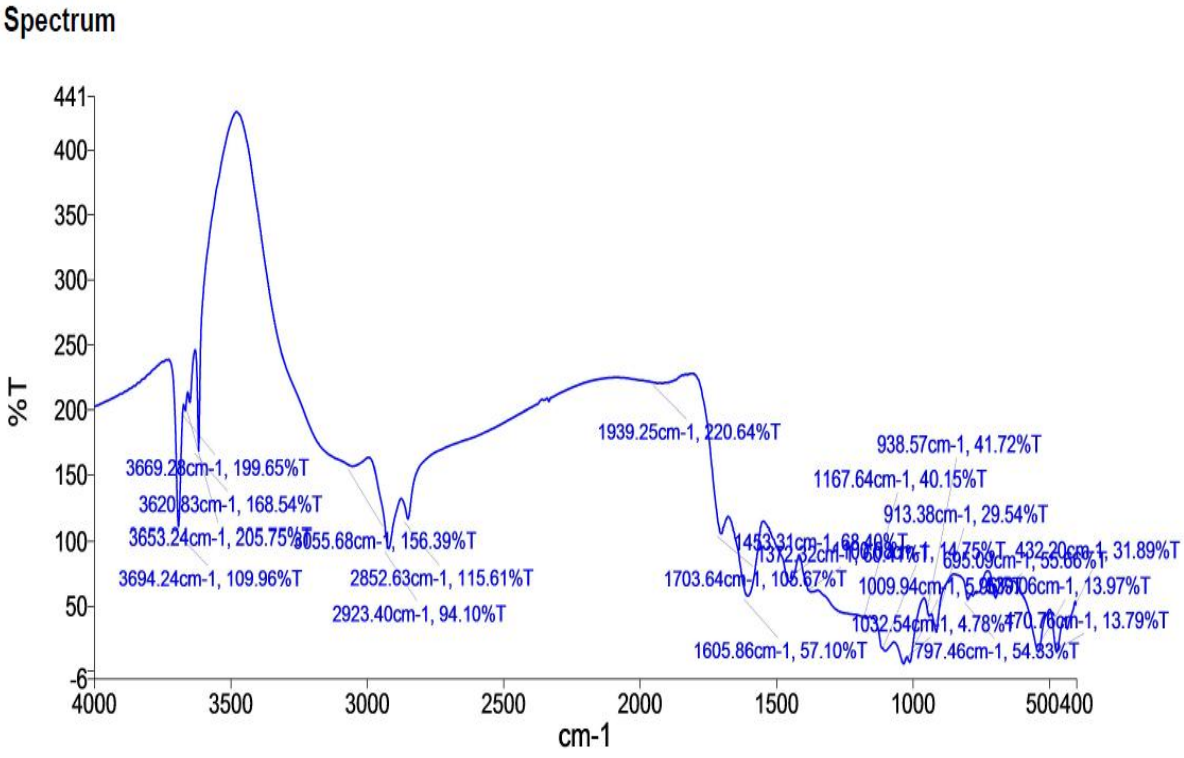

Figure A2-41: FTIR Transmittance spectra for MCL – 18

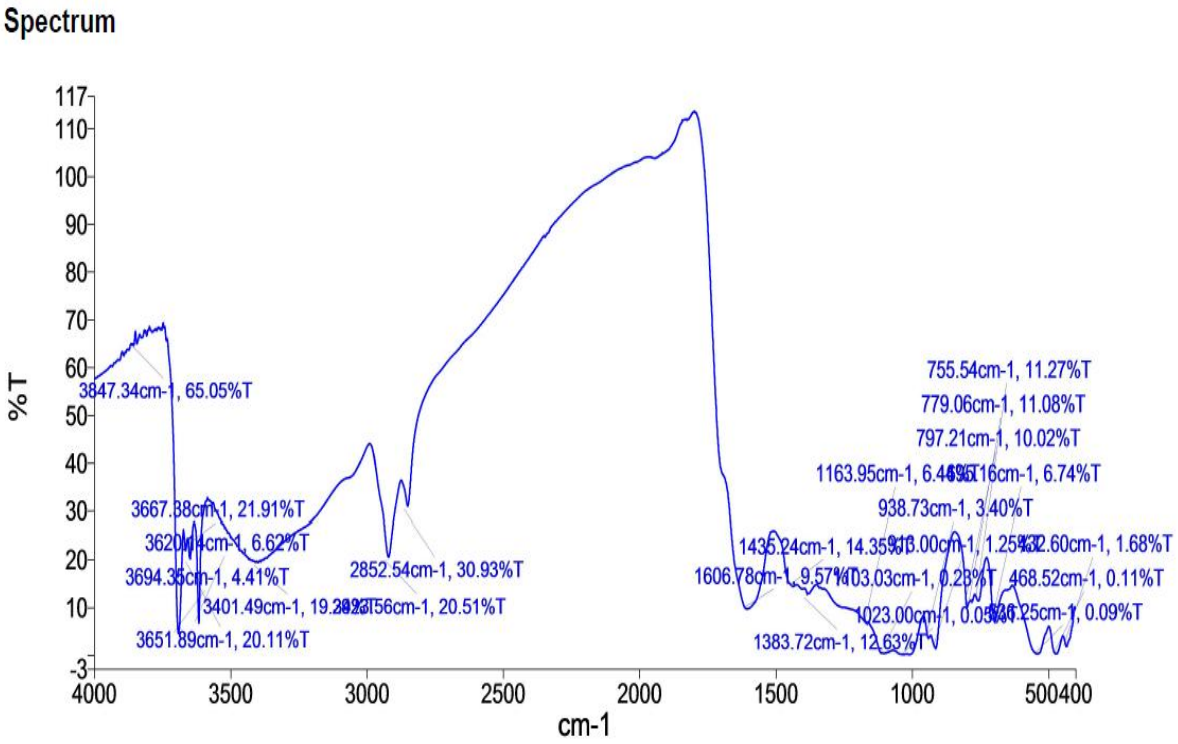

Figure A2-42: FTIR Transmittance spectra for MCL – 19

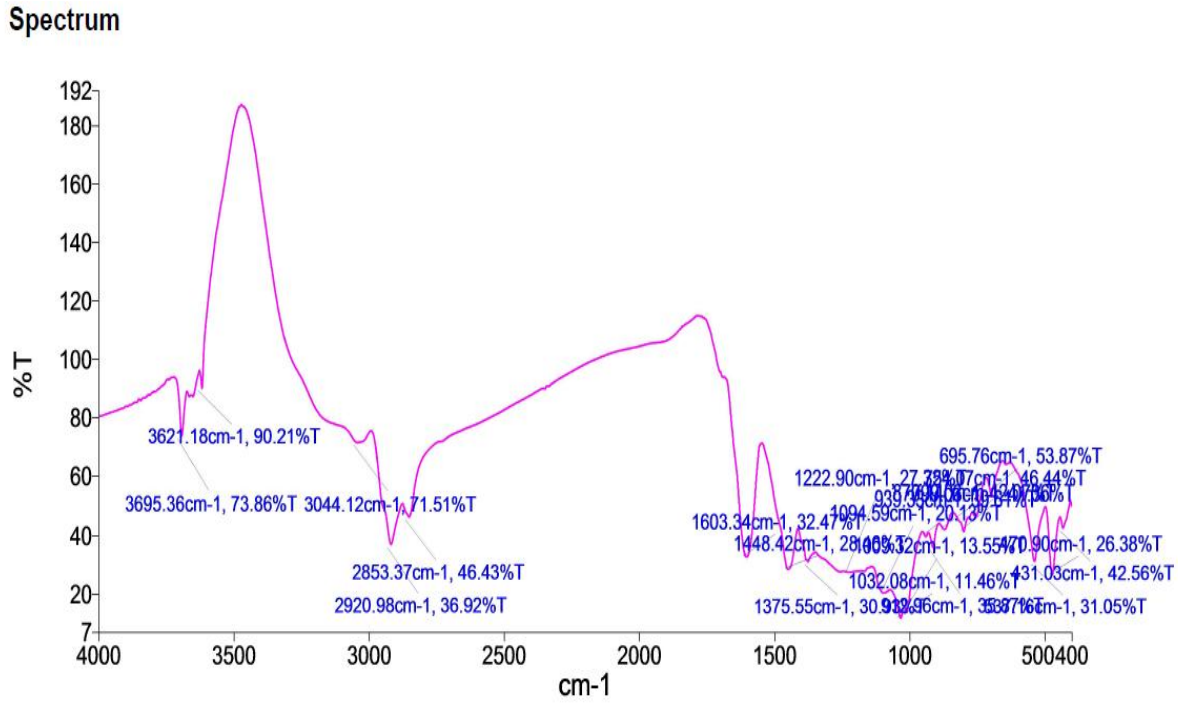

Figure A2-43: FTIR Transmittance spectra for BCCL-1

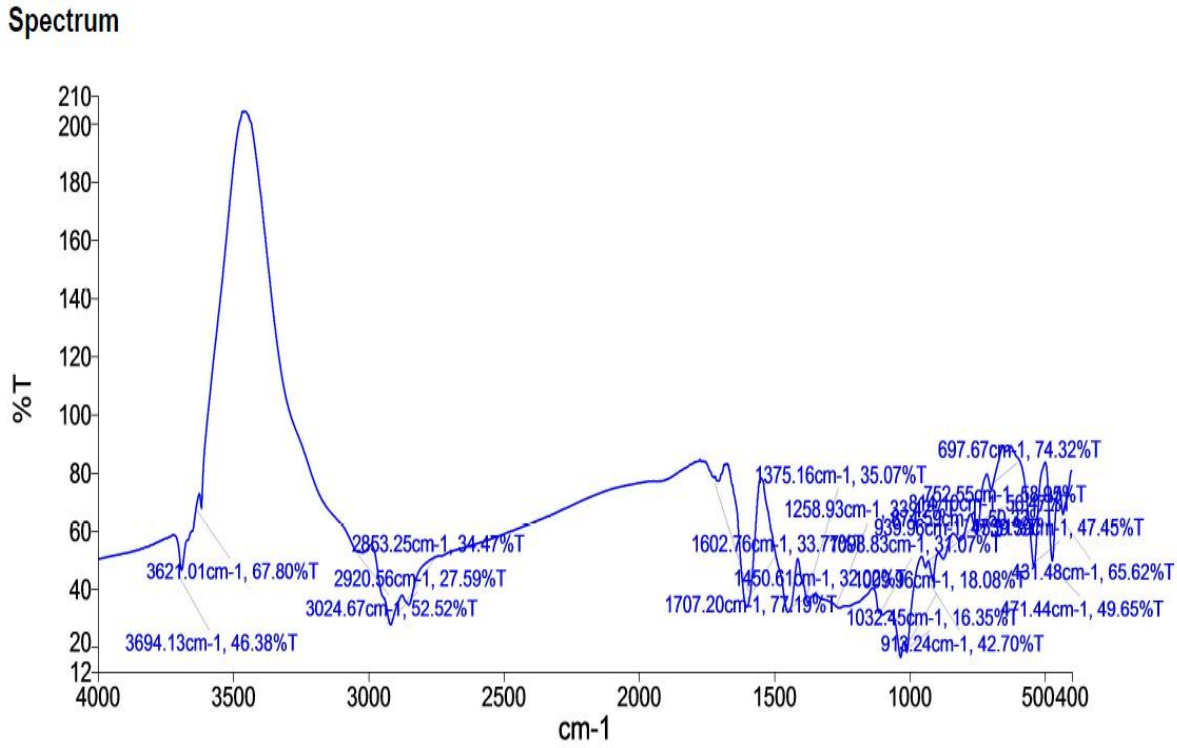

Figure A2-44: FTIR Transmittance spectra for BCCL-2

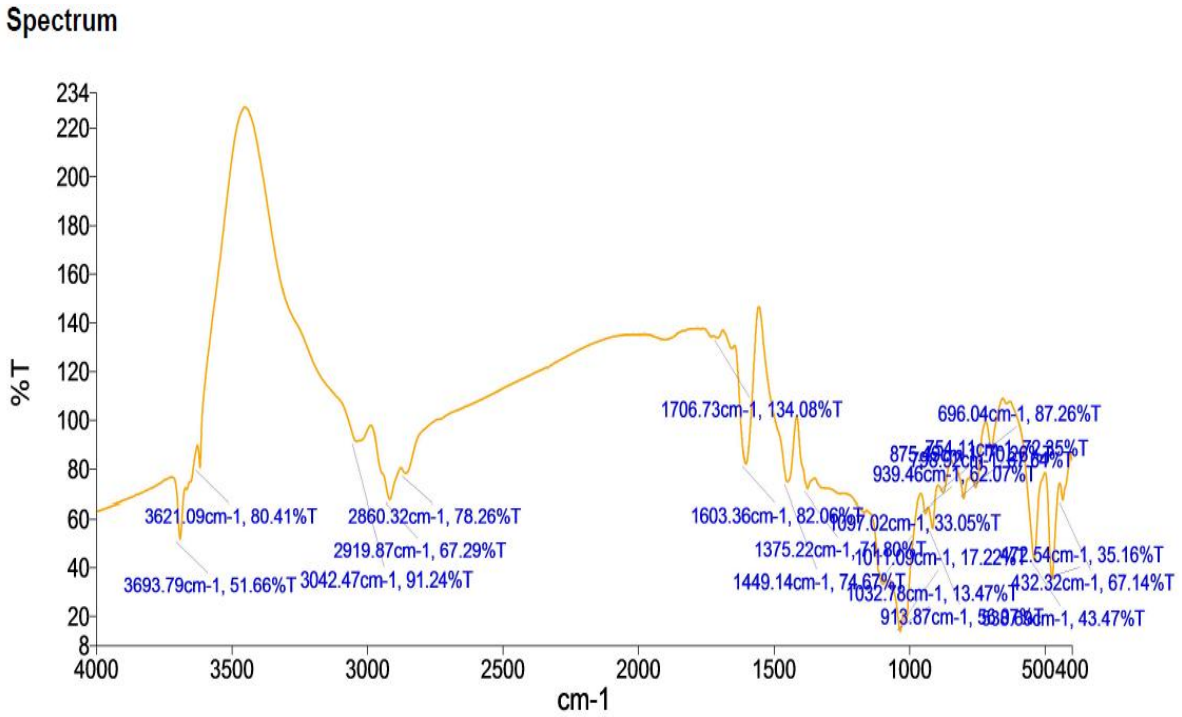

Figure A2-45: FTIR Transmittance spectra for BCCL-3

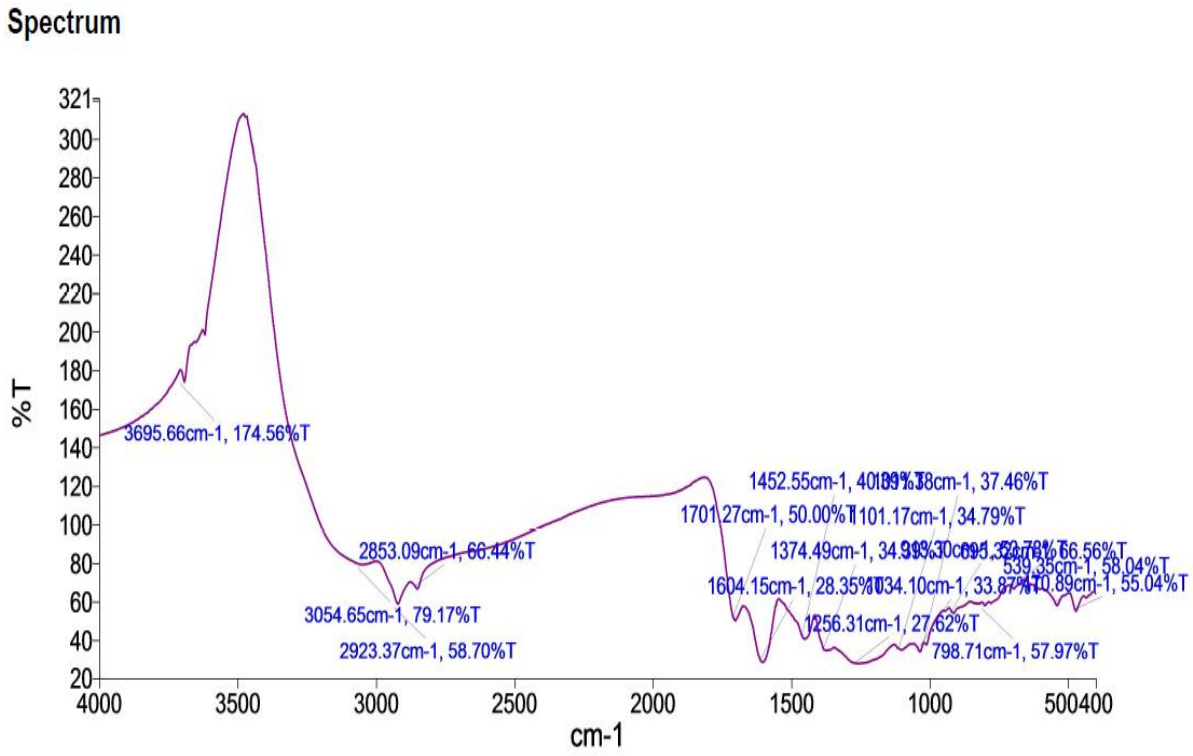

Figure A2-46: FTIR Transmittance spectra for WCL-1

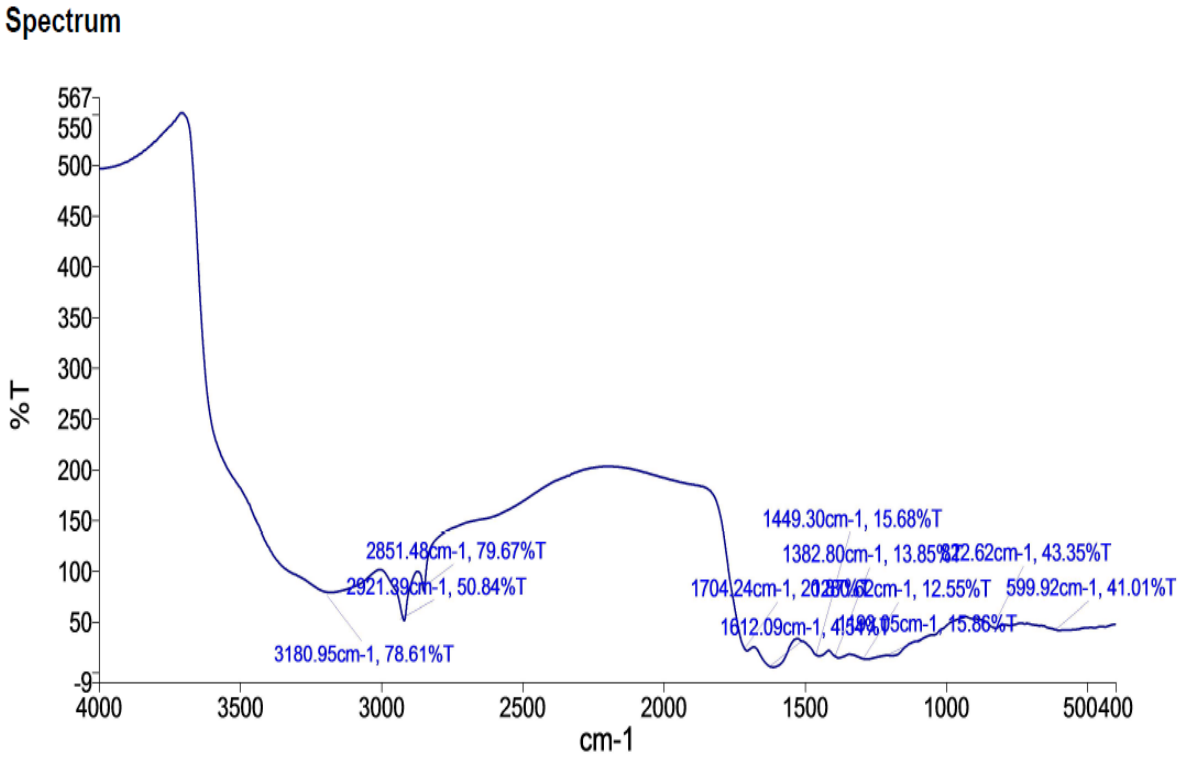

Figure A2-47: FTIR Transmittance spectra for NLC-1

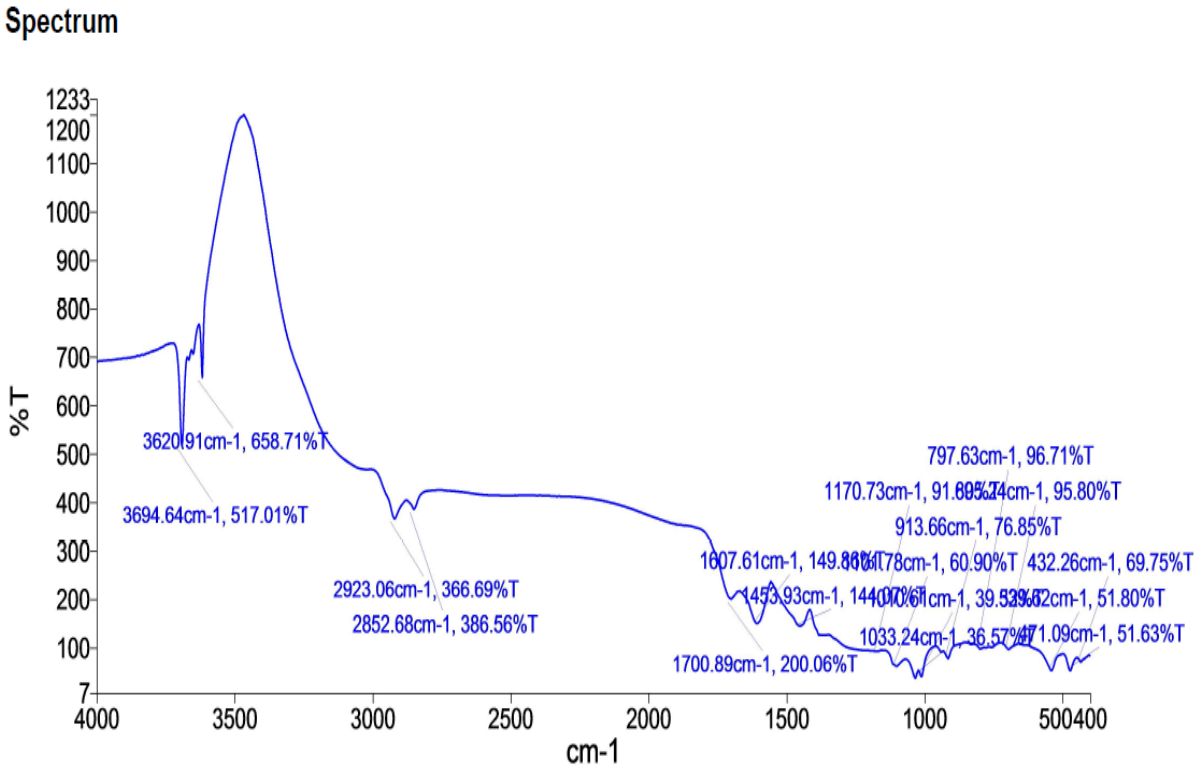

Figure A2-48: FTIR Transmittance spectra for NCL-1

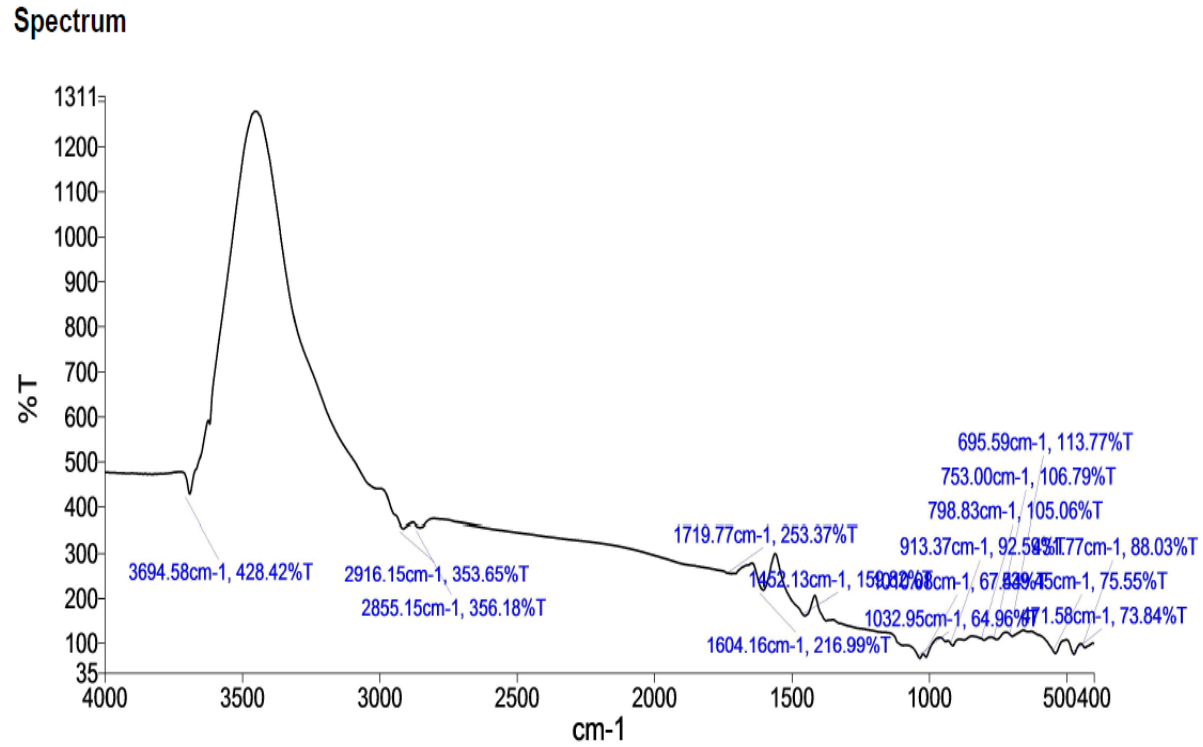

Figure A2-49: FTIR Transmittance spectra for NSW Coal Sample